出國報告(出國類別：研習)

美國參考物質製備之程序、技術及管理制度研習

服務機關：行政院環境保護署環境檢驗所

姓名職稱：巫月春　主任秘書

派赴國家：美國

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摘　要

本次研習前往美國國家標準技術研究所(National Institute of Standards and Technology , NIST)馬里蘭州蓋瑟斯堡（Gaithersburg）及南卡羅來納州查理斯頓市(Charleston)研習，透過與負責美國土壤或底泥基質之參考物質研發部門之量測實驗室化學科學部門無機化學計量組及統計相關科學家進行研討，以了解美國標準技術研究所對於環境土壤或底泥基質標準物質製備技術，及其參考物質化學成分濃度、均勻度、穩定度、保存、運送等評估規範與管理制度，作為國內環境品質管制或盲樣製備與管理之參考。此外，國內環境檢測實驗室常購買美國商業機構製備之品管樣品，作為人員能力評估或檢測品質管制樣品，此次亦將參訪美國 SIGMA-ALDRICH 及 ERA 兩家土壤及水質參考物質製備機構及能力測試提供商業機構，就其能力試驗商品製備、確認、結果評估與其產品說明文件等之技術實務進行較深入之研討，解開我們對其品管樣品濃度確認證書所提供之數據範圍之疑惑與誤用。

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壹、目的

本所常需購置國外參考物質（SRM）或驗證物質（CRM）作為各縣市環保局及檢測機構檢測能力之評估，但由於國內各地土壤組成不同，再加上土壤污染管制標準之限制，使得國際上能夠包含國內土壤基質及王水消化前處理之土壤類能力評估之參考物質（SRM）或驗證物質（CRM）不多，因此本所近期(自 104 至 106 年)規劃製備本土之土壤與底泥檢測能力評估之樣品，本次研習前往美國國家標準技術研究所參訪而有機會了解美國標準技術研究所環境標準物質技術資料及參考物質濃度驗證均勻度、穩定度、保存、運送等評估規範與管理制度，作為國內環境品質管製或盲樣製備與管理之參考。

此外，因國內環境檢測實驗室常購買美國商業機構製備之品管樣品，作為人員能力評估或檢測品質管制樣品，此次亦將參訪美國兩家土壤及水質參考物質製備機構及能力測試提供機構，就其能力試驗樣品製備、確認、結果評估與其產品說明文件等之技術實務進行較深入之研討，頓時解開了我們對該產品數據的疑義。

貳、過程

一、研習期間:105 年 9 月 14 日至 24 日

（一）105 年 9 月 14 日 起程赴美

桃園搭機至美國加州洛杉磯，轉機到科羅拉多州丹佛市，搭車至懷俄明州(Wyoming)拉勒米(Laramie)市

（二）105 年 9 月 15 日 拜訪 SIGMA-ALDRICH 公司

參訪議題：土壤及水質參考物質製備機構及能力測試提供者（PT Provider）運作實務

受訪人：Christopher Rucinski 等人

（三）105 年 9 月 16 拜訪 ERA 公司(科羅拉多州 Golden 市)

參訪議題：土壤及水質參考物質製備機構及能力測試提供者（PT Provider）運作實務

受訪人：Melissa L. Wright 等人

（四）105 年 9 月 17 日 搭機前往美國華盛頓特區

（五) 105 年 9 月 18 日 假日(整理資料)

（六) 105 年 9 月 19 日 參訪美國 NIST(Gaithersburg 辦公區）物質量測實驗室化學科學部門無機化學計量組(NIST, Inorganic Chemical Measurement Group, Chemical Sciences Division Material Measurement Laboratory)

參訪議題：環境參考物質量測技術與方法

受訪人：Dr. Michael R.Winchester 等人

（七) 105 年 9 月 20 日 繼續拜訪 NIST 物質量測實驗室化學科學部門無機化學量測組及資訊技術實驗室統計工程部門數學統計專家(Statistical Engineering Division Information Technology Laboratory Mathematical Statistician)

參訪議題：環境參考物質量測技術與方法及參考或確認物質(SRM/CRM)的統計及計算方法

受訪人：Dr. Michael R. Winchester 及 James Yen(統計)等人

（八) 105 年 9 月 21 日上午 搭機至南卡羅萊納州查理斯頓市(Charleston, South Carolina)

（九) 105 年 9 月 21 日下午及 9 月 22 日上午 拜訪 NIST 查理斯頓市辦公區化學科學部門環境化學科學組(NIST at Charleston, South Carolina Chemical Sciences Division Environmental Chemical Science)

參訪議題：土壤及底泥 SRM 製備

受訪人：Dr. Steven J. Christopher 等人

（十) 105 年 9 月 22 日下午 返程(查理斯頓搭機至紐約)

（十一) 105 年 9 月 23 日至 24 日 返程(紐約至桃園)

二、研習內容

(一) SIGMA-ALDRICH/RTC 公司

國內取得國際測試實驗室認證規範 ISO/IEC 17025 認證體系之環境實驗室所孰知的美國測試領域能力試驗機構 RTC 公司，係位於美國懷俄明州拉勒米市(Laramie, WY USA)，數年前與美國 SIGMA-ALDRICH 公司合併，併入後 RTC 公司成為 SIGMA-ALDRICH 區域部門，主要業務仍為辦理美國或美國以外之能力試驗及檢測品管樣品之製造與銷售。在美國環保署(USEPA)國家環境實驗室認證組織(The NELAC Institute,簡稱 TNI,其中 NELAC 為 National Environmental Laboratory Accreditation Conference 縮寫)能力測試提供者(Proficiency Testing Provider,簡稱 PT Provider)登錄名稱則為"MilliporeSigma" (TNI PT Provider Code 為 TNIPTP41)詳見附錄 1 及附錄 2。目前該部門(以下簡稱 RTC)員工人數為 28 人，年營業額 1200 萬美元。主要收入來自參加能力試驗者所繳費用及其所販售之品管樣品(即為經能力試驗後所剩樣品)。RTC 每年會辦理數次(一般約 4 次)符合 USEPA 或 ISO/IEC 17025 之美國國內或國際能力試驗，在辦理能力試驗前一年半即會在該公司網站上公布其能力試驗期程與項目資訊，接受美國國內或國外之測試實驗室報名參加能力試驗。測試時，各參與實驗室進行樣品分析期限為 45 天，RTC 會在收到各實驗室測試結果後 21 天內完成測試結果的統計分析，每次參加測試之實驗室家數最少要有 4~8 家，理想是 20 家以上。如果是美國國內能力試驗，參加能力試驗之實驗數家數可達百家；若參加的家數不足時，RTC 公司甚至會付費邀請某些實驗室參加測試，以符合統計要求。

RTC 美國國內能力試驗樣品製備及結果統計主要係依據 USEPA 規範之製備樣品及統計結果。USEPA 會具體規範各介質(例如飲用水、放流水、土壤、廢棄物…等)能力試驗樣品配製項目、濃度範圍及結果統計方法。RTC 通常每 3 個月會製備一批次能力試驗樣品，每批次樣品約有 300 瓶，同一次測試，樣品具有多批次，這些樣品寄發給付費參加能力試驗的實驗室測試後，依照 USEPA NELAC 統計規範或國際能力試驗規範 ISO 34 至 35 進

行統計分析，除將結果通知參加者外，各該批次所剩樣品即成為 RTC 之品管樣品(產品標示 CRM, Certificate Reference Material)。RTC 公司曾於 2003 年(92 年)與國內代理商和合作，由國內代理商提供國內台中及彰化地區重金屬污染場址之土壤，製備成能力測試樣品，這些樣品並由當時取得本署土壤重金屬許可之檢測機構 23 家檢驗室進行能力試驗，此測試後之樣品至今仍常被國內土壤實驗室做為技術或績效評估之品管樣品(CRM045-050 及 CRM046-050)。

RTC 公司所製備與銷售的品管樣品會提供此樣品之性質與應用、製備、分析、結果評估與其他資訊等證明文件(Certificate of Analysis ,簡稱 CoA)，以其編號 CRM026-050 之土壤品管樣品(附錄 3)為例，重點摘要說明如下：

1. 本樣品取自美國洛磯山脈輕微污染場址之天然土壤基質含砷、鎘、鉻、銅、鉛、汞、鎳及鋅等 8 種金屬之 CRM，樣品經風乾、過篩及均勻化處理後，每一瓶裝填重量約 50 克。此批樣品經 26 家檢驗室以 USEPA SW846 Method 3050 6010(ICP)及 7060(AA)檢測分析，分析濃度資訊則是依據 USEPA/AALA RM-03 及 ISO 34 至 35 進行統計分析之結果。
2. 樣品經均勻度測試濃度確認，取樣時須將樣品混合均勻後再稱取 1 克，精秤至 0.1 克，分析結果並以乾基計算。
3. 結果評估: 確認參考值係以 USEPA SW846 Method 7060A 分析砷、USEPA SW846 Method 7471B 分析汞、王水消化 DIN 38414-S7 分析鎘、鉻、銅、鉛、鎳及鋅等 6 種重金屬元素進行實驗室間測試，統計方法是 USEPA BIWEIGHT Method，信賴區間為 95%。

前述所舉樣品常為國內環境測試實驗室引用於真實樣品分析時之品管樣品或本所辦理檢測機構之許可管理時之技術能力評估樣品，長期來我們採用之合格範圍為其 CoA 上之 Confidence Interval(CI)，本次參訪研習時，特別針對這些數據請教 RTC Site Manager－Christopher Rucinski 先生，釐清 CoA 上之 Confidence Interval(CI)是依據 USEPA NELAC 能力試驗提供者在試驗樣品「Assigned Value」研究之品質管制規範(附錄 6)，USEPA NELAC 對此之規範為 1 倍相對標準差以內，RTC 公司將此 CI 顯示於報告中主要

是展現本 CRM 樣品製備時之濃度確認分析數據品質之良莠；26 家國際實驗室能力測試之結果，以 Grubb Test 方法去除離群值(Outliner)，採用 Z-SCORE 統計方法得到之範圍列為 CoA 上之 Prediction Interval(PI) 即為一般的 3 倍 SD，這個回答終於解開我們長期以來對合格範圍很窄及 CI 如何而來的困惑。另外，此次 Christopher Rucinski 也介紹 RTC 在樣品製備上，除直接採自然受污染場址之天然基質樣品，也會採用外添加污染物方式，其添加技術以有機農藥樣品為例，將農藥標準品溶於丙酮中，再添加於過 100 mesh 篩網之天然土壤樣品，標準溶液量需要蓋過土壤，將樣品至於排煙櫃，將丙酮慢慢吹乾即可得到濃度均勻分布之有機農藥之土壤基質樣品。這個製備方法將可提供國內未來發展含基質盲樣之參考，也是本次參訪重要收穫。

圖 1　參訪位於美國懷俄明州拉勒米市(Laramie, WY) 的土壤及水質參考物質製備機構及能力測試商業機構 SIGMA-ALDRICH/RTC 公司，主要訪談對象為 Christopher Rucinski (Site Manager 右 1) 及 Patrick Brumfied (Product Manage 右 2)

(二) ERA 公司(Environmental Resource Associates, Inc.)

ERA 公司也是國內取得國際測試實驗室認證規範 ISO/IEC 17025 認證體系之環境實驗室孰知的美國測試領域能力試驗機構，它位於美國科羅拉多州 Golden 市，數年前將美國另一家性質相同的製造與銷售品管樣品公司 APG 併入，幾年前又再與另一家儀器公司-WATERS 公司合併。合併後 ERA 公司還是以辦理能力測試及品管樣品之製造銷售業務為主，在 USEPA 國家環境實驗室認證組織(TNI)能力測試提供者(PT Provider) 登錄名稱則為 " Environmental Resource Associates, Inc." (TNI PT Provider Code 為 TNIPTP21)詳見附錄 1 及 5。ERA 公司規模較 RTC 大，員工人數為 74 人，主要營業項目也是舉辦美國國內及國際能力試驗及其品管樣品販售 (亦為經能力試驗後所剩樣品)。ERA 公司每年辦理數次、類別及批次皆比 RTC 公司多且廣，幾乎是每個月均有辦理，這些資訊均可以從該公司網站上查得(目前可以查到 2018 年初的能力測試計畫)。參加測試之實驗室家數最少要求為 7 家，ERA 公司有一項較快速提供結果之能力試驗，其最快可以在參加者將檢測數據送達之 10 天內給統計分析報告(這種測試 ERA 稱為「QUICK PT」，它是利用經能力測試過之所剩之樣品與統計結果，來評估 QUICK PT 參加者之能力試驗結果)，其測試相關規範亦符合 USEPA 或 ISO/IEC 17025 之美國國內或國際能力試驗。

USEPA NELAC 對能力試驗提供者所配製基質、配製濃度範圍及能力測試合格範圍均有明確規範。詳見附錄 4~5(飲用水、放流水)。表列各規範說明如下：

1. 「Conc Range」即是能力試驗樣品須配製之濃度範圍，以附錄 4 表之飲用水中銅為例，測試樣品中銅的配製濃度範圍應介於「50-2000」 ug/L 間。
2. 「Acceptance Criteria」之「a」及「b」是 NELAC 根據過去一段時間各能力試驗公司所配置樣品配製濃度結果統計出不同濃度經由線性回歸分析得到配製濃度值之截距(b)與斜率(a)；「c」及「d」根據過去能力測試，在

規定濃度範圍內各樣品測試結果之 SD 統計出不同濃度經由線性回歸分析得 SD 之截距(c)與斜率(d)。實際應用情況，例如：某公司配製廢水中 100 ug/L 的砷，由表 5 查得其 a=0.9916，b=1.2647，c=0.0422，d=5.1741 此時預估配製之平均濃度為 100 ug/L＊0.9916＋1.2647＝100.4 ug/L，100.4＊0.0422＋5.1741＝9.4%(此即為 1SD 的範圍) 3＊9.4%＝28.2% 能力試驗之 Acceptance Criteria 即為 100.4＋28.2%至 100.4－28.2%。一般 NELAC 對飲用水 Acceptance Criteria 訂為 2SD，放流水為 3SD，甚至有些項目 NELAC 即規定一個固定百分比之範圍，例如飲用水中銅測試樣品濃度範圍應介於「50-2000」ug/L 間其能力測試合格範圍均為配製濃度之 10%。

3.「NELAC PTRL」是指 NELAC 要求參加能力測試的實驗室回報極限值，意思是若能力測試樣品製備時並未添加此項目標物，實驗室回報超過 PTRL 之數據，即顯示該檢驗室有背景污染問題。以飲用水中砷為例，測試樣品製備時若未添加砷標準品，檢驗室回報樣品砷之濃度應小於 3.5 ug/L，大於 3.5 ug/L 時，顯示受測試之檢驗室應檢討系統中砷污染來源。

ERA 公司製備樣品採外添加方式，其土壤樣品基質是採取自科羅拉多州內較乾淨(未受污染)的土壤，將其研磨至 20~100 mesh 後，再外添加金屬或其他標準品製備成待測樣品。經能力測試過之樣品即成為 ERA 公司其另一項產品－品管樣品，該公司稱為 RM(Reference Material)，本所較常使用其水質有機項目之樣品，本次參訪就其 CoA 文件上相關參數之意義進行討論，以其 WatR™ Pollution Volatiles 710(如附錄 6)為例，說明如下：

1.「Certified Value」一般是配製值或 USEPA Assigned Value（即前述之預估配製之平均濃度)其中「Acetone」為「<3.9 ug/L」意思是指本樣品中並未添加 Acetone，測試時此樣品之 Acetone 含量檢測值應該小於 3.9 ug/L（此即為 NELAC PTRL）。

2.「QC Performance Acceptance Limits」是 ERA 公司根據該公司此項目過去測試累積資料庫中濃度、基質(若是土壤樣品粒徑一會納入考量)

相同或相似者之統計範圍，提供給檢測者進行內部品質控管之參考。

3.「PT Performance Acceptance Limits」指此批號樣品進行能力測試之結果可被接受範圍，此範圍係依據國際或 NELAC 規定所統計或計算而得。

4.「Proficiency Testing Study」之「Mean」、「Recovery」及「n」是指 ERA 公司本身進行能力試驗計畫所執行之樣品測值之平均值、回收率及實驗次數等。

5.「NIST Traceability」之「SRM Number」及「Recovery」為 ERA 公司於能力試驗計畫時併同分析濃度相近 NIST 的 SRM 所得回收率數據，主要係佐證該公司的確認值(Certified Value)品質之良莠。

圖 2 參訪位於美國科羅拉多州 Golden 市的另一家土壤及水質參考物質製備機構及能力測試樣品公司 ERA 公司(其為 WATERS 公司之子企業，辦公室門口招牌掛的是 WATERS；訪談對象為 Melissa L. Wright (Senior Account Manager 右 3)及 Michael Blades (Technical Manage 左 1，他目前兼任 NELAC 能力試驗委員會委員)

(三) 美國 NIST 材料量測實驗室化學科學部門無機化學量測組（Inorganic Chemical Metrology Group, Chemical Sciences Division Material Measurement Laboratory）及資訊技術實驗室統計工程部門數學統計專家（Statistical Engineering Division Information Technology Laboratory Mathematical Statistician）

美國國家標準與技術研究所（NIST）成立於 1901 年，現為美國商務部的所屬研究機關，主要任務為藉由加強量測科學、標準與科技之發展，提升美國企業創新能力與強化產業競爭力，進而達成強化經濟安全與提升生活品質的目標，總部位於馬里蘭州的蓋瑟斯堡（Gaithersburg），另外有部分設施則在科羅拉多州的博爾德市（Boulder）。職員人數約有 3000 名，NIST 所設立的實驗室亦廣納來自全球各地從事研究工作的專家，因此在蓋瑟斯堡辦公區之人數有六七千人。NIST 主要有 5 個實驗室、2 個中心及 3 個外部計畫組成，分別為物理測量實驗室（Physical Measurement Laboratory, PML）、通信技術實驗室（Communications Technology Laboratory, CTL)、工程實驗室 Engineering Laboratory, EL）、資訊技術實驗室（Information Technology Laboratory, ITL）、材料測量實驗室（Material Measurement Laboratory, MML）、中子研究中心（Center for Neutron Research, NCNR）、奈米科學技術中心（Center for Nanoscale Science and Technology, CNST）及 Baldrige 績效卓越計畫（Baldrige Performance Excellence Program）、Hollings 製造業拓展夥伴（Hollings Manufacturing Extension Partnership, MEP）、進階製造辦公室（Advanced Manufacturing Office, AMO)等，其組織架構圖如附錄 7。其中材料測量實驗室是 NIST 兩個計量實驗室之一，該實驗室負責化學、生物和材料科學方面的國家參考實驗室。業務範圍從工業、生物和環境材料和製程的組成、結構和性質之基礎和應用科學研究到開發和傳輸工具，包括參考測量程序，認證參考物質等。目前可提供超過 1300 項具有良好特性(物質之物理、化學或生物特性)的組成或性質的標準參考物質，作為儀器校準或開發新的測量方法所需。材料測量實驗室部門下有 8 個單位，分別為應用化學材料部（Applied Chemicals and Materials Division）、生物分子測量部門（Biomolecular Measurement Division）、生物系統和生物材

料部門（Biosystems and Biomaterials Division）、化學科學部門（Chemical Sciences Division）、物質測量科學部門（Materials Measurement Science Division）、材料科學與工程部門（Materials Science and Engineering Division）、數據與資訊辦公室（Office of Data and Informatics）及參考物質辦公室（Office of Reference Material）等單位。其中化學科學部門主要產品和服務是品質保證計畫及參考物質，部門再細分成：化學資訊研究組（Chemical Informatics Research Group）、化學程序與核測量組（Chemical Process and Nuclear Measurements Group）、環境化學科學（Environmental Chemical Sciences Group）、環境樣品儲存組（Environmental Specimen Bank Group）、氣體感測計量組（Gas Sensing Metrology Group）、無機化學量測組（Inorganic Measurement Science Group）、有機化學量測組（Organic Chemical Measurement Science Group）等。其無機測量組負責 pH、電分析、X 射線、光學和質譜以及傳統分析方法開發，無機和有機金屬物質的鑑定和測量技術評估與應用。此次主要拜訪對象為無機化學量測組主管同仁及資訊技術實驗室（ITL）之統計工程專家，透過實驗室參觀與人員訪談討論方式，藉此了解美國環境參考物質特性量測技術方法、統計計算及確認等議題之運作實務。

ISO Guide 30 規範參考物質（Reference Material, RM）是指相對於一種或多種特定性質足夠均勻和穩定的材料，其已被確定為適合其在測量過程中的預期用途。RM 在性質上可以是定量或定性，例如物質或物種的身份，用途可以包括測量系統校準、測量程序的評估、作為另一物物質之指定值或品質管制。確認參考物質（CRM）則是參考物質之特徵在於對一個或多個指定性質的計量有效的程序，伴隨有提供指定特性數值及不確定性和計量上可追溯性陳述之證書。據此，NIST 對其標準參考物質（Standard Reference Material, SRM）亦有進一步定義，摘要如下：

1. **NIST Standard Reference Material®（SRM）**：由 NIST 發布符合認證標準且附證書或分析證書，報告其特性結果及適當使用的資訊之 CRM，具有良好的性能，使用最先進的測量方法或技術來確定化學組成和/或物理性質。使用 SRM 有三個主要目的：（1）幫助開發準確的分析方法（2）校準

用於促進貨物交換的測量系統，進行品質控制，確定性能特性或在最先進的極限測量性能（3）確保測量品質保證計畫的長期完整性。而”NIST Standard Reference Material® （SRM)”包含“SRM”的菱形標誌，在美國是有專利和商標局註冊的。

2. **NIST Reference Material(RM)**：由 NIST 發布具有調查報告(不是證書）作為（1）科學或技術研究（2）確定原形参考物質之功效（3）提供均勻和穩定的物質使不同實驗室的研究者能夠確保他們正在研究相同物質(4）確保在由 NIST 以外的組織生產和認證的物質被定義為符合公共利益使用。NIST 的 RM 可以滿足 ISO 的 RM 與 CRM 定義（取決於生產的組織)。
3. **NIST Traceable Reference Material ™（NTRM™)**：一種商業化生產的参考物質，與現有 NIST 標準化學測量具良好追溯性；通過 NIST 定義的標準和協議建立連接可追溯性，以滿足要服務的計量，符合這些要求的參考物質生產廠商允許使用 NTRM 商標。NIST NTRM 可以被監管機構認為等同於 CRM。
4. **NIST Certified Value**：NIST 對 SRM 證書或分析證書報告數值之準確性具有最高自信，因為所有已知或可疑的偏差來源已經被 NIST 充分調查或計算。
5. **NIST Reference Value**：在 NIST 證書、分析證書或調查報告中提供其真實值之最佳估計，其中對所有已知或可疑之偏差來源未有完整調查。
6. **NIST SRM Certificate or Certificate of Analysis**：NIST SRM 證書是包含物質名稱、物質描述和預期目的文件、美國商務部標章、認證機構名稱、物質使用與儲存說明、量測不確定度、檢測方法、有效期限及其他技術被認為是正確使用的必要資訊等等。
7. **NIST RM Report of Investigation**：NIST 發行之 RM 文件，內容包括物質正確使用之技術資訊、美國商務部的標章以及授權發布之 NIST 官員姓名與職稱。此種文件沒有提供 NIST 的認證值，報告的內容的作者可能是由 NIST 以外的組織之成員。

8. **NIST Report of Analysis (ROA)**：NIST 分析報告內容包括所使用之基礎材料、SRM 之製造和認證方法程序、外部合作對象、物質之使用說明、特殊包裝、處理和儲存說明，以及穩定性測試計劃。通常 ROA 僅供內部 NIST 使用。

表 1 NIST 化學品 SRM 或 RM Value-Assignment 數據品質評估分類

Mode		Certified Value	Reference Value	Information Value
1	Certification at NIST Using a Single Primary Method with Confirmation by Other Method(s)	Y		
2	Certification at NIST Using Two Independent Critically-Evaluated Methods	Y	Y	
3	Certification/Value-Assignment Using One Method at NIST and Different Methods by Outside Collaborating Laboratories	Y	Y	
4	Value-Assignment Based On Measurements by Two or More Laboratories Using Different Methods in Collaboration with NIST		Y	Y
5	Value-Assignment Based on a Method-Specific Protocol		Y	Y
6	Value-Assignment Based on NIST Measurements Using a Single Method or Measurements by an Outside Collaborating Laboratory Using a Single Method		Y	Y
7	Value-Assignment Based on Selected Data from Interlaboratory Studies		Y	Y

在美國工業、學術界或政府機關使用 NIST SRM 來促進商業、貿易、研究與開發。目前 NIST 的 SRM 可用於工業材料生產與分析、環境分析、健康測量、科學和計量學等基本測量領域。此外，NIST 出版了許多有關 SRM 開發、分析與使用之文獻資料可供各界參考。2000 年發行的『NIST Special Publication 260-136 Standard Reference Materials Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements』（如附錄 8）。表 1 為 NIST 依量測方法將其化學品的 SRM 或 RM 之指定值(value-assigning)數據品質成分 NIST Certified Value、NIST Reference Value 及 NIST Information Value 等 3 個等級。「NIST Certified Value」表示 NIST 在其準確性具有最高自信的數據，即所有已知的或疑似來源的偏差已經被 NIST 充分調查或解釋；「NIST Reference Value」是 NIST 對真實值做了最佳估計，其中所有已知或疑似來源的偏差並沒有得到 NIST 充分調查。「NIST Information Value」是對 SRM / RM 使用者感興趣和使用的數值，但用於評估與該值相關的不確定度資訊不足。

在用於化學品的 SRM 的 Value-Assignment 歸類模式是基於 NIST 專業人員過去對特定基質或分析目標物的經驗與知識、當時測量能力及分析方法的數據品質、量測結果及物質的預期用途等因素決定。SRM 的量測數值最終指定 Certified Value、Reference Value 或 Information Value 都基於特定值歸類模式以及所得數據相對於此物質的預期用途的品質評估。提供 SRM 認證值的技術和方法要經過嚴謹的評估並且該基質分析之準確性經證明、評估和解決這些方法的潛在誤差來源“基於比率”（儀器使用檢量線）的方法，使用高純度，良好表徵的主要參考化合物或物種作為其校準的基礎(直接或通過重量分析法校準溶液，例如 NIST 元素溶液 SRM)。NIST 對於量測方法及其測試的細節均詳細記錄於其內部分析報告（NIST Report of Analysis)，並且經常在該領域的技術期刊中發表。當採用外部實驗室的量測驗證結果時，由 NIST 化學科學技術實驗室（CSTL）負責選擇外部實驗室，並對這些報告數據進行技術評估。無論是在 NIST 內部還是由任何外部合作實驗室執行確認，若有來自其他國家計量

實驗室合適的 SRM 或 CRM 可用時，必須一併進行 RM 或 CRM 的分析。各類模式使用時機：

1. Certification at NIST Using a Single Primary Method with Confirmation by Other Method(s)
    a. 該方法與該基質之所有潛在的重要誤差來源都已明確評估，即在分析報告中提供了可能且合理的其他誤差來源以及為什麼在這種特殊情況下不期望它們具有顯著性。
    b. 以另一個方法進行確認，而確認的方法至少須通過下列條件之一，但原級方法（Primary Method）和任何確認方法之間的一致性要求，必須在實驗計畫中先明定：(1)檢測其他基質與濃度相似之 SRM 或 CRM（2）NIST 認為適當的第二種技術（3）選擇具有經驗之外部合作實驗室的測量結果。

2. Certification at NIST Using Two Independent Critically-Evaluated Methods

NIST 使用兩個或多個精確評估的獨立方法，方法獨立性很重要，雖然很少有兩種分析方法具有完全不同的誤差和變異性來源，但須選擇誤差源最顯著不同的方法， 例如：

a. 選擇盡量減少樣品製備和最終測量步驟的方法。

b. 依產生的不同的物理、光譜或化學現象定量的方法。

c. 所選擇的方法/程序對樣品基質中感興趣的分析物具有符合需求的精密度與準確度。

d. 對確認所使用方法間的要求須先確認，並記錄於實驗計畫中。

3. Certification/Value-Assignment Using One Method at NIST and Different Methods by Outside Collaborating Laboratories

在 NIST 沒有合適的第二獨立方法的情況時，須仔細選擇外部合作實驗室進行確認的過程。理想情況下，這種合作始於實驗設計過程的開始，NIST 和外部實驗室分析人員都能夠協調測量的細節、數據分析和報告要求，並注意以下事項：

a. NIST 的方法和外部合作實驗室的方法，必須有經過嚴謹評估並證明在樣

品基質條件下，可提供準確的結果。

b. 外部合作實驗室使用的方法，應不同於 NIST 所使用的方法。

c. 外部合作實驗室的數據報告應包括實驗計畫及不確定度重要來源評估資訊。

4. Value-Assignment Based on Measurements by Two or More Laboratories Using Different Methods in Collaboration with NIST

在 NIST 沒有合適量測方法的情況下，以兩個或更多個與 NIST 合作實驗室使用不同方法進行測量所得。此模式可以提供 SRM NIST Reference Value 或 Information Value。此模式之要求：

a. 外部合作實驗室的方法對此基質之準確性經證明。

b. 外部合作實驗室提供的分析涉及至少兩種不同的方法（見表 1 Mode 2）。

c. 除非大量實驗室/方法提交數據，否則外部合作實驗室的數據報告應包含足夠的資訊，以評估所有重要的不確定性來源。

5. Value-Assignment Based on a Method-Specific Protocol

在方法定義參數的情況下，感興趣參數的值來自於使用經協議的適當方法。適當實施此模式需要：

a. 所使用經認可適用於測量該基質之分析物種（或性質）的方法。

b. 只使用來自經驗豐富的實驗者的數據。

c. 使用 NIST 或外部實驗室協議的特定方法進行測量。

d. 特定方法的 Value-Assignment，需要有三個以上對方法有豐富經驗的參加者。

6. Value-Assignment Based on NIST Measurements Using a Single Method or Measurements by an Outside Collaborating Laboratory Using a Single Method

在某些情況下，使用者的預期用途不需要 NIST 認證值作為指定值。此模式可提供 NIST Reference Value 或 Information Value。

a. 使用 NIST 模式 2 的方法中使用的方法(Two Independent NIST Methods)，即該方法可能在過去已經被用作 SRM 認證的幾種方法之一，但在這種情

況下是唯一使用的方法。

b. 外部實驗室使用的方法須證實能夠在此基質中能提供適當的精密度和準確度。

c. 外部協作實驗室的數據報告應包含足夠的資訊，以評估重要的不確定性來源。

7. Value-Assignment Based on Selected Data from Interlaboratory Studies

此模式允許 NIST 利用以參考物質的 value-assignment 為的目外之而實驗室間量測研究。在此模式下：

a. 特定研究必須有良好的文件記錄，並由信譽良好的單位來組織。

b. NIST 的化學科學和技術實驗室負責評估分析程序的適當性，以確定 value-assignment 所選用之數據。

本次參訪過程 Dr. Michael R.Winchester 特別安排我們進一步了解其 Primary Method 之儀器與技術，包括 XRF、INAA、ICP、ICP-MS、ID-ICP-MS、滴定法(採重量法，即滴定時不以體積計算)、庫倫計等等，這些方法有的是基礎量的量測方法，有的是 NIST 專家自行研究能確認其量測不確定度之各貢獻來源，或此量測技術經發表或參加國際比測所得結果良好，專家對此檢測方法技術所得之確認值非常有信心。過程中只要我們對 Primary Method 有任何疑義，他們就立刻可以提供很多他們在國際研討會議或期刊上發表之研究論文，顯見他們不是自我感覺良好而已。例如 XRF 方法，若該元素有 NIST 的參考物質，專家們即利用高溫熔融法將參考物質與 $LiBO_2$ 於高溫加熱熔融打錠，製成 5 至 6 個不同濃度之檢量線，進行定量分析，且每加入一個元素即做一次檢測定，再利用數學模式計算基質干擾效應，因此他們認為 XRF 方法所有檢測過程之誤差，均可以被控制，所以把 XRF 歸為 Primary Method。另一個有趣的事是 NIST 負責滴定法的專家(Tom)，在滴定分析時不是體積法而是採重量法，即滴定時，以精密天平來秤取前後重量差計算其濃度，因為他認為體積法誤差較大，且秤重時以泡泡紙包覆滴定溶液瓶(隔熱)方式稱取重量，他發現手部操作秤重時中，手中溫度會影響滴定溶液秤重量，顯見其實是非常注重這些分析的誤差小細節，再次證實其數據的準確度不是僅來自於其自信心。對於

SRM 文件中所提供之分析數據報告分類模式除參照前述 7 種模式進行分類外，NIST 負責此 SRM 計畫之專家仍有主觀判斷之空間，甚至有的 SRM 第二驗證確認實驗室是美國政府另一個研究機關－美國地質調查局（USGS）所執行，NIST 專家依然認為無法歸類 Certified Values，但無論如何，此 SRM 文件中所列數據歸類為 Certified Values 、Reference Value 或 Information Value 均會在其『Report of Analysis 』中敘明(Report of Analysis 應該記錄之內容詳見本報告的 11 頁)。參訪時並以 NIST SRM 2709a 為例進行討論(其 Certificate of Analysis 文件如附錄 9)，訪談紀要如下：

1. 這批號 SRM 規劃為 5 年需求量，由 NIST 委託 USGS 採集 California San Joaquin 地區 3 個場址的休耕地農業土壤，每個場址採集面積約 4m^2，採集時先去除表層植被，共採 20 桶(每桶容量為 5 加侖之樹膠桶)送入 USGS 實驗室，在室溫下乾燥後篩分，移除大於 2mm 之粗顆粒後所剩土壤約 50 kg，分批以球磨機進行研磨(≥95%的粉末通過 200 mesh 篩網)及 V 型混合器混合均勻及輻射滅菌後裝瓶，每瓶含有 50 克，共製備 2650 瓶。
2. USGS 於每 100 個瓶為批次進行一次均勻性測試，測試方法為 X 射線熒光光譜法（WD-XRF）、感應耦合電漿發射光譜法（ICP-OES）、感應耦合電漿質譜法（ICP-MS）等方法，NIST 也進行均勻性評估。
3. 樣品分析：使用 XRF 及中子活化法(INAA)評估瓶裝 SRM 中所選擇的元素的均勻性。在少數情況下，由統計學觀察到均勻度有顯著差異，對於大多數元素，SRM 不均勻性的估計相對標準偏差≤1%、鈣約為 2%，鉻約為 3%，汞有較顯著的不均勻性(詳見該 COA 之表 2)。在 NIST（Gaithersburg，MD）和 USGS（Denver，CO）進行該 SRM 的分析結果如該 COA 之表 1、表 2 和表 3 所示之 Certified Values、Reference Values 及 Information Values。用於每個的分析技術元素列於該 COA 之表 4，包括 XRF、INAA、PGAA、ICP-OES、ICP-MS、ID-ICP-MS、CV-ID- ICP-MS、CCT-ICP-MS 等方法；分析人員列在該 COA 之表 5 和表 6 中。確認分析時整批取 6 個樣品，每個樣品進行二重覆分析，使用 2 個獨立方法時，每個方法取 6 個樣品，每個樣品進行二重覆分析。

3. Certified Values：共有鋁等 19 種元素，如該 Certificate of Analysis 文件之該 COA 之表 1。除鎘及鉛外，所有元素的 Certified Value 是兩個或三個分析結果平均值，。此 Certified Values 基於嚴謹獨立分析技術所得的結果，NIST 對其準確度具有最高自信心，認為其中所有已知或可疑的偏差來源已經調查或考慮了。訪談中發現，鎘和鉛這個元素 NIST 只用 ID/ICP/MS 單一方法檢測即歸類在 Certified Value，並不符合前述 1~3 的歸類模式，經詢問 Dr. Michael R. Winchester 表示，鉛的歸類是由負責此專案的專家主觀判斷，專案專家認為 ID/ICP/MS 方法檢測結果是非常確定而有高度自信認為正確無誤，因其已經對所有不確定性來源進行了完整的評估。當下隨即指著實驗室牆上所貼著國際研討會所發表之技術論文及國際比對之優異結果，佐證其自信心是有所本的。相對的，也有部分元素雖經 USGS 獨立方法確認，卻不歸在 Certified Values 群，而是歸在 Reference Values 群，個人非常好奇問，難道 NIST 不相信 USGS 的分析數據？Dr. Michael R. Winchester 想了一下，回答因為 USGS 的分析結果報告沒有量測不確定度數據。

4. Reference Values 共有砷等 15 種元素，係由 NIST 以單一方法分析所得，Reference Values 是對真實值的最佳估計值，提供之量測不確定度可能未包括所有來源；其中，汞的 Reference Value 係取 6 瓶樣品以 ID-CV-ICP-MS 分析結果之平均值表示。結果顯示其有不均勻之情況；濃度範圍從 0.8mg/kg 至 1.1mg/kg。

5 Information Values 有硼等 10 種元素，由 NIST 以單一方法分析所得，這些值並沒有提供量測不確定度之數值。

6.這批土壤樣品 NIST 並提供以 US EPA 認可之 8 個土壤檢測合約實驗室以 Methods 200.7 and 3050B 進行溶出程序以混合酸消化或熔融檢測之 Certified Values、Reference Values 及 Information Values，其中 7 個使用 USEPA 方法 200.7;其餘實驗室使用 USEPA SW-846 方法 3050B 用於土壤樣品的製備，以 ICP-OES）測定各元素含量，8 個實驗室中有 6 個提供了重複分析數據，這些結果以平均值作統計。結果如該 COA 之表 A1。（這種州政府或聯邦 EPA 需求的溶出試驗方法，其確認證書是以附錄方式提供元素的測值，稱之為

Procedure defined）

值得一提的是 NIST 在 Gaithersburg 有一座核子反應爐，因此中子活化分析技術幾乎是他們大部元素的定量方法，反應爐之中子經由 D_2O 冷卻照射樣品，讓樣品受中子撞擊活化後，產生連鎖反應，在不同時間下量測樣品中各元素 γ 射線強度定量樣品，這也是為何這麼多研究人員匯集在 Gaithersburg 辦公區作研究之原因。也因此造成 Gaithersburg 辦公區空氣中鉛的背景值較其他地區高的緣故，因此數年前 NIST 將一部分重金屬 SRM 製備業務移至南卡羅萊納州查理斯頓市(Charleston, South Carolina)的原因之ㄧ。

圖 3 位於馬里蘭州蓋瑟斯堡（Gaithersburg）NIST 總部，本次參訪的材料量測實驗室化學科學部門無機化學量測組位於該辦公區 Building 227。

圖 4　位於中間的是材料量測實驗室化學科學部門無機化學量測組 Supervisory Research Chemist Leader － Dr. Michael R.Winchester

圖 5 與資訊技術實驗室統計工程部門數學統計專家（Statistical Engineering Division Information Technology Laboratory Mathematical Statistician）討論參考物質濃度確認之統計問題；右 1 是 Chief Statistician－Dr. Antonio Possolo，右 3 是 Dr.James Yen，他 3 歲時由台灣移民美國，略聽得懂台語

圖 6 在 NIST 馬里蘭州蓋瑟斯堡（Gaithersburg）總部餐廳附近園區的這顆蘋果樹，是源自於牛頓發現萬有引力的那顆樹，樹上長了許多蘋果果子，果子比一般蘋果小很多，大小約比葡萄大一點。樹下設有桌椅，只是不知是不是期待有另一個牛頓出現

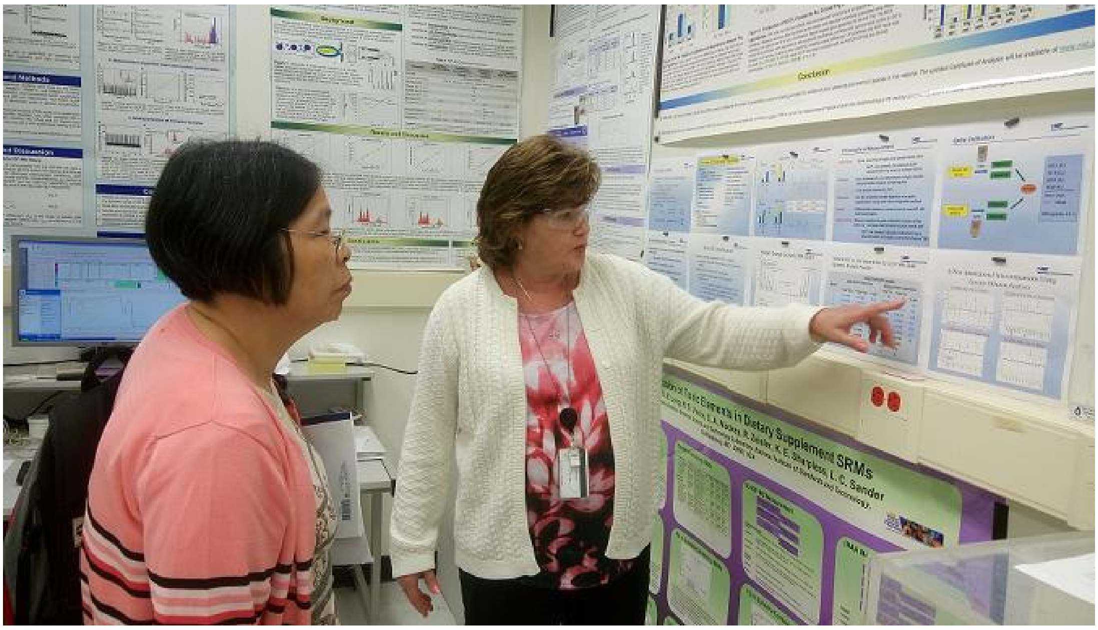

圖 7　NIST 材料量測實驗室化學科學部門無機化學量測組的專家們，對其參考物質成份確認值之分析技術非常有自信，實驗室牆上貼滿他們發表期刊論文，參加國際研討會或國際比測結果良好的壁報

(四) 美國 NIST 查理斯頓市辦公區化學科學部門環境化學科學組(NIST at Charleston, South Carolina Chemical Sciences Division Environmental Chemical Science)

美國 NIST 查理斯頓市辦公區在 South Carolina Charleston,和美國 NOAA（National Oceanic and Atmospheric Administration）、South Carolina 州政府單位及 2 個州立大學等 5 個單位合署辦公。NIST 這個辦公區重要工作內容是動物基因或物種研究與保存，研究人員約有 30 人。此行主要拜訪對象是化學科學部門環境化學科學組 Dr. Steven J. Christopher 等人，參訪議題為土壤及底泥 SRM 製備。目前這個辦公區負責的製備的 SRM 共有 8 個土壤（7 個已完成，1 個研製中）及 4 個底泥。8 個土壤基質 SRM 編號分別為 2700、2701、2709a、2710a、2711a、2586、2587 及 2706(研製中)；4 個底泥基質 SRM 為 1646a、1944、2702 及 8704 等，其中 2700 與 2701 為六價鉻污染場址製成之土壤 SRM，前者為低濃度後者高濃度 ；2709a、2710a 與 2711a 為土壤基本元素含低中高濃度不同之 SRM(a 表是同一批 SRM 重製)；2586 與 2857 為鉛污染場址之製成之土壤 SRM，前者濃度較低約 500ppm，後者為 3000 ppm ；1646a 為河口底泥，1944 為河川底泥之 PAHs、PCBs、PBDE、PCNs、HBCDs 及元素之有機及無機成分 SRM，2702 為海洋底泥中無機成分 SRM，8704 為河川底泥之元素成分 SRM。此行主要想了解 NIST 查理斯頓辦公區之 SRM 製備規劃與濃度確認之操作實務，這些 SRM 主要配合 USEPA 污染管制檢測品管需求而研製，現地採集之土壤均交給 USGS 進行研磨、過篩等均質化後交給 NIST，此外 USGS 也執行濃度確認工作；底泥參考物質則與軍事或 NOAA 等相關機關合作研製；這些合作機關與 NIST 均屬契約的夥伴關係;此外他們還有非環境基質之 SRM,例如蘋果汁、海產類基質等。在土壤或底泥 SRM 製備採取混樣方式製成，每個編號研製時皆規劃製成 2500 瓶左右的量，進行均勻度測試時，取 20 瓶以 WD－XRF 進行元素檢測分析，每瓶進行二重覆，計算均勻度，另外也進行不同取樣量之均勻度檢測。有機成分進行均勻度確認時，並非每個成分都做檢測，僅選擇代表性成分進行檢測，例如 PCBs 檢測時選與內標化合物相同之 131 進行

確認，但檢測時每一個成分圖譜均經人工進行定性定量確認，所使用之技術與方法則與前述 Gaithersburg 所使用的相同，以 primary method 為主(附錄10)。基本上一個 SRM 製備規劃到完成約需 3 年時間，因此通常都規劃 5~10 年市場需求量。

圖 5 NIST 位於查理斯頓市之辦公廳舍，並 NOAA、South Carolina 州政府單位及 2 個州立大學等 5 個單位合署辦公

參、心得

一、 美國商務部所屬 NIST 進行參考物質之製備驗證，透過合約實驗室機制將土壤或底泥參考物質之研磨過篩均勻化等前處理及濃度確認工作，交給對土壤或底泥物化性質專業更了解之美國地質研究所執行，兩個單位合作無間各展所長，可以讓國家資源的效益充分發揮。但最後由 NIST 負責計劃的專業科學家以其專業權威判斷參考物質之等級，顯示可以看得出其對技術專業之信任與尊重。

二、 NIST 參考物質之檢測結果依量測方法將其化學品的 SRM 或 RM 之指定值(value-assigning)數據品質成分 NIST Certified Value、NIST Reference Value 及 NIST Information Value 等 3 個等級，各等級之依檢測方法、不確度分類之規範明確，使用者可以依其使用目的選擇最適等級之參考物質。

三、 本次參訪 NIST 之土壤及底泥參考物質製備相關部門時間雖然短暫，但藉由與關鍵科學家訪談方式，對於 NIST 的土壤或底泥基質參考物質製備規劃、現地採樣、篩分、研磨、均勻度與濃度確認、取樣檢測數目與方法、檢測技術及統計方法規範及後續參考物質監管等，相關實務操作細節有較深入的討論與了解，對本所未來土壤和底泥盲樣研製具有相當參考價值。

四、 NIST 參考物質之製備與成分驗證結果，除提供工業及貿易用途之外，亦會配合美國環保署污染管制需求，另外以附錄提供該參考物質以美國環保署污染管制規定的酸消化方法執行檢測數據之結果，此不相隸屬之部署間展現出其行政協調、相互支援之一體政府效能，頗讓我佩服。另外，美國在經歷 911 攻擊事件後，政府機關門禁管制加嚴，NIST 辦公區管理十分嚴格，此次參訪美進出辦公室建築物都有門禁管制，就連餐廳都不例外，但是他們也提供非常友善的服務，例如這次前往參訪接洽過程，Dr. Michael R. Winchester 要我提前上網申請進入園區，因此當日在門口登記處早已幫我把來賓證製作完成，幾分鐘內我就順利進入園區；在 NIST 網站可以查得辦公區和附近旅館間接駁車及旅館收費；園區午餐、下午茶時

間收費等等，這些細小的事務，對遠到而來的訪客或研究的人員是十分貼心友善的服務。

五、 此行特別長途跋涉到懷俄明州及科羅拉多拜訪 SIGMA-ALDRICH 及 RTC 兩家能力試驗機構，藉由與土壤及底泥能力試驗相關人員訪談方式，了解其水質、土壤或底泥能力試驗樣品製備、濃度確認所依照的規範是來自美國 EPA，得取得兩家公司能力試驗辦理週期、項目資訊，甚至對國內常使用這兩家公司所販售之品管樣品，分析證書所提供各項資訊細節非常深入的討論，進而解開長久以來我們對這些樣品資訊之疑惑與誤用，已值回票價。另外，參訪過程兩家相關人員提供許多美國環保署 NELAC 組織之能力試驗規範資料，其中有一位是 NELAC 能力試驗委員會委員，對於 NELAC 能力試驗相關規範之訂定細節十分了解，更讓此行充實而豐碩。

肆、建議

一、 此次參訪兩家商業能力試驗機構，對其辦理國際或美國國內能力試驗規劃、樣品製備與確認、時程安排、結果處理過程有更多的了解，建議未來國內自行製備之土壤或底泥能力試驗樣品，可以透過這兩家或其他美國環保署認可之商業能力試驗機構辦理國際性能力測試，各批次能力試驗所剩樣品即可以此國際能力測試結果統計數據作為國內能力試測合格與否之判斷依據。

二、 NIST 或兩家商業能力試驗機構以外添加方式製備能力測試樣品，使得製備樣品之濃度較多元，對此外添加方式的了解雖然很粗淺，但對國內未來以土壤或底泥基質外添加有機污染物成分之能力試驗樣品的方向是可行的。

三、 美國 EPA 所屬的 NELAC 組織對於能力試驗樣品配製濃度與合格範圍經過多年資料累積統計回歸分析得到，對於該組織之能力試驗之規範或其他資訊的收集研析，應有助於本所辦理檢測機構水質、土壤、底泥及廢棄物

等類別之績效評鑑之參考，建議應更進一步收集其相關資訊，另外 NELAC 對於環境實驗室之認證所擔任的角色與本所相近，未來亦可尋求參訪 NELAC 組織之機會，對於該組織之環境實驗認證、評鑑及監督制度規範研訂之背景沿革與組織運作等等有更深入之瞭解，提供國內環境檢測機構之許可與管理制度精進之參考。

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PT Providers

A provider of PT samples must be accredited by a Proficiency Testing Provider Accreditor (PTPA) that meets the TNI requirements. A listing of organizations that are accredited to provide PT samples is available below.

TNI PT Provider Code	PT Provider	Contact	City, State	Phone
TNIPTP16	Absolute Standards, Inc.	Stephen Arpie	Hamden, CT	800-368-1131
TNIPTP19	Advanced Analytical Solutions, LLC	Frederick Anderson	Parkersburg, WV	304-422-4274
TNIPTP21	Environmental Resource Associates, Inc.	Christopher Crone	Golden, CO	303-431-8454
TNIPTP41	MilliporeSigma	Patrick Brumfield	Laramie, WY	307-742-5452
TNIPTP31	NYS DOH Environmental Laboratory Approval Program	Kenneth Aldous	Albany, NY	518-474-7161
TNIPTP36	NSI Solutions, Inc.	Mark Hammersla	Raleigh, NC	800-234-7837
TNIPTP46	Phenova	Jason Campbell	Golden, CO	303-940-0033

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ANALYTE CONCENTRATIONS

Element	Reference Value	S.D.	Confidence Interval	Prediction Interval
Arsenic, As	5.41	2.49	4.64 - 6.19	0.481 - 10.3
Cadmium, Cd	12.9	1.81	12.2 - 13.6	9.10 - 16.7
Chromium, Cr	36.9	13.9	31.6 - 42.1	7.77 - 65.9
Copper, Cu	22.5	2.83	21.5 - 23.6	16.6 - 28.5
Lead, Pb	30.7	6.73	28.1 - 33.4	16.7 - 44.8
Mercury, Hg	2.42	0.320	2.16 - 2.46	1.30 - 3.32
Nickel, Ni	19.3	4.38	17.6 - 21.0	10.1 - 28.4
Zinc, Zn	169	18.4	161 - 176	130 - 207

All values are expressed in mg/Kg (parts per million) on a dry weight basis. The Reference Values were determined by using USEPA SW846 Method 7060A for Arsenic, by using USEPA SW846 Method 7471B for Mercury, and by using Aqua Regia DIN 38414-S7 Method for Cadmium, Chromium, Copper, Lead, Nickel, and Zinc.

Confidence Interval (C.I.) range is the 95% C.I. for the Reference Value. The Prediction Interval (P.I.) is the 95% P.I. around the Reference Value. Measurements should fall within the P.I. range 19 of 20 times. The Certified Reference Values were established through extensive interlaboratory testing. All values were calculated using the USEPA BIWEIGHT Method.

"THIS PRODUCT WAS DESIGNED, PRODUCED, AND VERIFIED FOR ACCURACY AND STABILITY IN ACCORDANCE WITH USEPA/AALA RM-03 AND ISO GUIDES 34 AND 35."

Certifying Officer

RTC
2931 Soldier Springs Road
Laramie, WY 82070
Phone: 800.576.5690 or 307.742.5452
Fax: 307.745.7936
Web: www.RT-Corp.com

PRODUCT INFORMATION

Instructions For Use
Metals on Soil CRM026-050
Certified Reference Material

● Caution: Please read instructions before using

1. Scope and Application

The Metals on Soil Certified Reference Material (CRM) sample consists of a single sample jar containing approximately 50 grams of material. This soil is from a slightly contaminated site located in the Rocky Mountain Region of the US, and is not "spiked or fortified" in any manner. The matrix was air dried, sieved to remove extraneous debris, and homogenized. Being a real-world waste sample the analyst is challenged by the same preparation problems, analytical interferences, etc. as is typical for similar matrices received by the laboratory for analysis.

Rigorous analyses identified, quantified, and certified several metals which are listed on the enclosed Certificate of Analysis. The sample has been analyzed by a minimum of twenty laboratories in a round-robin to meet the requirements specified by the EPA/AALA RM-03, ISO 34 and 35. The sample was certified by USEPA SW846, 3rd edition Method 3050, 6010 (ICP) and 7000 series (AA) methods. The sample is suitable for use by these and other similar methods.

2. Sample Preparation

The entire sample lot has been tested and certified for inter-sample homogeneity, Due to potential settling and stratification in storage, shipping and handling the sample ***must be thoroughly mixed*** as stated in the method. If the reference method is being used, weigh out 1 gram of sample to the nearest .01 gram.

Weigh out additional material to determine percent moisture. All values reported on the Certificate of Analysis are reported on a ***dry weight*** basis.

3. Analysis

Follow the analysis instructions given in the referenced method. If there are any technical questions/problems encountered, or difficulties experienced in the use of these samples, please contact:

R.T. Corporation
Technical Support
Tel. (307) 742-5452
Fax (307) 745-7936

4. Evaluation of Results

The Reference Value, 95% confidence interval(C.I.) for the Reference Value and 95% Prediction Interval around the Reference Value were obtained by the methods identified on the Certificate of Analysis. Samples were selected in a random fashion from the beginning to the end of the bottling sequence and sent for analysis to a 26 laboratory round-robin. The data produced in the round-robin was used to calculate reference values by the USEPA EMSL-CINN's computer program "BIWEIGHT".

The Biweight program generates a "Student's t" like statistic for constructing confidence intervals on data sets that may have heavier tails than a classical Gaussian distribution. This is appropriate for symmetric, stretch-tailed curves that are often encountered in analysis of homogenous samples via interlaboratory studies. The Biweight method is also more robust in handling data that results from determinations at or near the method detection limits. For data sets that are Gaussian, the Biweight estimates are comparable to traditional calculation methods.

The generated Biweight mean, Biweight standard deviation and Biweight standard deviation of the mean are used to calculate the 95% Confidence Interval (CI) for the mean and the 95% Prediction Interval (PI). For normally distributed data, the Biweight 95% CI compares well to the classical calculation method used to generate a 95% CI. For non-Gaussian data sets, the Biweight method is more robust in data treatment.

Biweight data are also used to calculate a 95% PI. The 95% PI compares well to a 95% tolerance limit calculated using classical methods. For normally distributed data, the Biweight 95% PI typically represents approximately a ±2 Biweight standard deviation window around the Biweight mean. Again, the Biweight method is more robust than classical methods when handling non-Gaussian data sets.

Laboratories performing the same analytical procedures on a sample whose values have been determined by the Biweight method can assume that the true mean, as determined by the method, is within the 95% CI window. Laboratories analyzing the sample should have results within the 95% PI window 19 out of 20 analyses.

Additional information on the program may be obtained by referring to the reference or by downloading the program from the EMSL-CINN bulletin board.

Additional analytes detected, but not certified, are listed in parenthesis and can be used to determine detection and approximate values.

5. Informational Values

pH – 4.29

Texture Classification – Sandy Loam

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[1] Kafadar, K, A Biweight Approach to the One-Sample Problem, Journal of the American Statistical Association, Vol. 77, No. 378, June, 1982, pp. 416-424

NELAC PT for Accreditation

Fields of Proficiency Testing with PTRLs

Drinking Water

Effective January 3, 2012

Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte[1,2]	Conc Range	Acceptance Criteria[3,4,5,6] a	b	c	d	NELAC PTRL[7]
			Red = Previous Experimental Analytes	Blue = New Analyte/Header/Footnote				Magenta = Changes	
			Microbiology	CFU/100 mL					CFU/100 mL
Drinking Water	0254	2500	Total Coliform[8,9,10]		Nine out of ten correct with no false negatives				Not Applicable
Drinking Water	0255	2530	Fecal Coliform[8,9,10]		Nine out of ten correct with no false negatives				Not Applicable
Drinking Water		2525	E.coli[8,9,10]		Nine out of ten correct with no false negatives				Not Applicable
				CFU (MPN)/mL					CFU (MPN)/mL
Drinking Water	0258	2555	Heterotrophic Plate Count (MF, PP)[11]	5 to 500	Log transform Mean ± 2 SD				2
Drinking Water	0258	2555	Heterotrophic Plate Count (MPN)[12]	5 to 500	Log transform Mean ± 2 SD				2
				CFU (MPN)/100 mL					CFU (MPN)/100 mL
Drinking Water		2525	E.coli (MF)[11]	20 to 200	Log transform Mean ± 2 SD				2
Drinking Water		2525	E.coli (MPN)[12]	20 to 200	Log transform Mean ± 2 SD				2
Drinking Water	0255	2530	Fecal Coliform (MF)[11]	20 to 200	Log transform Mean ± 2 SD				2
Drinking Water	0255	2530	Fecal Coliform (MPN)[12]	20 to 200	Log transform Mean ± 2 SD				2
Drinking Water	0254	2500	Total Coliform (MF)[11]	20 to 200	Log transform Mean ± 2 SD				2
Drinking Water	0254	2500	Total Coliform (MPN)[12]	20 to 200	Log transform Mean ± 2 SD				2
			Trace Metals	µg/L					µg/L
Drinking Water	0235	1000	Aluminum	130 to 1000	± 20% at < 500 ± 15% ≥ 500 fixed acceptance limit				104
Drinking Water	0140	1005	Antimony	6 to 50	±30% fixed acceptance limit				4.2
Drinking Water	0001	1010	Arsenic	5 to 50	±30% fixed acceptance limit				3.5
Drinking Water	0002	1015	Barium	500 to 3000	±15% fixed acceptance limit				420
Drinking Water	0141	1020	Beryllium	2 to 20	±15% fixed acceptance limit				1.7
Drinking Water	0226	1025	Boron	800 to 2000	±15% fixed acceptance limit				680
Drinking Water	0003	1030	Cadmium	2 to 50	±20% fixed acceptance limit				1.6
Drinking Water	0004	1040	Chromium	10 to 200	±15% fixed acceptance limit				8.5
Drinking Water		1045	Hexavalent Chromium (VI)	5 to 50	±20% fixed acceptance limit				4.0
Drinking Water	0091	1055	Copper	50 to 2000	±10% fixed acceptance limit				45
Drinking Water	0284	1070	Iron	100 to 1800	± 20% at < 250 ± 15% ≥ 250 fixed acceptance limit				80
Drinking Water	0005	1075	Lead	5 to 100	±30% fixed acceptance limit				3.5
Drinking Water	0236	1090	Manganese	40 to 900	±15% fixed acceptance limit				34
Drinking Water	0006	1095	Mercury[13a]	0.5 to 10	±30% fixed acceptance limit				0.35
Drinking Water	0237	1100	Molybdenum	15 to 130	±15% fixed acceptance limit				13
Drinking Water	0142	1105	Nickel	10 to 500	±15% fixed acceptance limit				8.5
Drinking Water	0007	1140	Selenium	10 to 100	±20% fixed acceptance limit				8.0
Drinking Water	0008	1150	Silver	20 to 300	±30% fixed acceptance limit				14
Drinking Water	0143	1165	Thallium	2 to 10	±30% fixed acceptance limit				1.4
Drinking Water	0238	1185	Vanadium	50 to 1000	±15% fixed acceptance limit				42
Drinking Water	0239	1190	Zinc	200 to 2000	±15% fixed acceptance limit				170
			Nutrients	mg/L					
Drinking Water	0009	1810	Nitrate as N	3 to 10	±10% fixed acceptance limit				2.7
Drinking Water		1820	Nitrate + Nitrite as N	3 to 10	±15% fixed acceptance limit				2.6
Drinking Water	0092	1840	Nitrite as N	0.4 to 2	±15% fixed acceptance limit				0.34
Drinking Water	0261	1870	Orthophosphate as P	0.5 to 5.5	±15% fixed acceptance limit				0.43

NELAC PT for Accreditation
Fields of Proficiency Testing with PTRLs
Drinking Water
Effective January 3, 2012

			Red = Previous Experimental Analytes	Blue = New Analyte/Header/Footnote				Magenta = Changes	
Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte[1,2]	Conc Range	Acceptance Criteria[3,4,5,6]				NELAC PTRL[7]
					a	b	c	d	
			Minerals	mg/L					mg/L
Drinking Water	0287	1575	Chloride	20 to 160	±15% fixed acceptance limit				17
Drinking Water	0010	1730	Fluoride	1 to 8	±10% fixed acceptance limit				0.90
Drinking Water	0145	2000	Sulfate	25 to 250	±15% fixed acceptance limit				21
Drinking Water	0286	1125	Potassium	10 to 40	±15% fixed acceptance limit				8.5
Drinking Water	0029	1155	Sodium	12 to 50	±15% fixed acceptance limit				11
Drinking Water	0283	1035	Calcium	30 to 90	±15% fixed acceptance limit				26
Drinking Water	0285	1085	Magnesium	2 to 20	±15% fixed acceptance limit				1.7
Drinking Water	0025	1550	Ca Hardness as $CaCO_3$	75 to 225	±15% fixed acceptance limit				64
Drinking Water		1755	Total Hardness as $CaCO_3$	83 to 307	±15% fixed acceptance limit				71
			Inorganic Disinfection By-Products	µg/L					µg/L
Drinking Water	0193	1535	Bromate	7 to 50	±30% fixed acceptance limit				4.9
Drinking Water	0260	1540	Bromide	50 to 300	±15% fixed acceptance limit				42
Drinking Water	0194	1570	Chlorate	60 to 180	±30% fixed acceptance limit				42
Drinking Water	0195	1595	Chlorite	100 to 1000	±30% fixed acceptance limit				70
			Misc Analytes	mg/L					mg/L
Drinking Water	0027	1505	Alkalinity as $CaCO_3$/L	25 to 200	±10% fixed acceptance limit				22
Drinking Water	0253	1520	Asbestos	1.5 to 20 MF/L	study mean		0.2971	0.4164	1 MF/L
Drinking Water		1620	Corrosivity	-4 to +4 SI units	± 0.4 SI units fixed acceptance				Not Applicable
Drinking Water	0146	1645	Cyanide, Total[13b]	0.1 to 0.5	±25% fixed acceptance limit				0.075
Drinking Water		1710	Dissolved Organic Carbon (DOC)	1.3 to 13	0.9744	0.0960	0.0402	0.0700	1.1
Drinking Water		1895	Perchlorate	4 to 20 µg/L	±20% fixed acceptance limit				3.2 ug/L
Drinking Water	0026	1900	pH	5 to 10 units	± 0.2 units fixed acceptance limit				Not Applicable
Drinking Water	0022	1945	Residual Free Chlorine	0.5 to 3.0	1.0000	0.0004	0.0776	0.0246	0.37
Drinking Water		1990	Silica as SiO_2	5 to 75	±15% fixed acceptance limit				4.2
Drinking Water	0288	1610	Specific Conductance	130 to 1300 µmhos/cm	±10% fixed acceptance limit				117 µmhos/cm
Drinking Water		2025	Surfactants - MBAS	0.1 to 1.0	0.9804	0.0054	0.0673	0.0348	0.020
Drinking Water		1940	Total Residual Chlorine	0.5 to 3.0	1.0000	-0.0048	0.0723	0.0065	0.40
Drinking Water	0024	1955	Total Filterable Residue	100 to 1000	±20% fixed acceptance limit				80
Drinking Water	0263	2040	Total Organic Carbon	1.3 to 13	±20% fixed acceptance limit				1.0
Drinking Water	0023	2055	Turbidity[13c]	0.5 to 8 NTU	0.9755	0.0593	0.0565	0.0661	0.36 NTU
Drinking Water		2060	UV 254 Absorbance	0.05 to 0.7 cm-1	0.9919	0.0043	0.0872	0.0034	0.038 cm-1

NELAC PT for Accreditation

Fields of Proficiency Testing with PTRLs

Drinking Water

Effective January 3, 2012

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Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte[1,2]	Conc Range	Acceptance Criteria[3,4,5,6]				NELAC PTRL[7]
					a	b	c	d	
			Volatile Organic Compounds (VOCs)[1]	µg/L					µg/L
Drinking Water	0039	4375	Benzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0037	4455	Carbon Tetrachloride	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0049	4475	Chlorobenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0054	4610	1,2-Dichlorobenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0041	4620	1,4-Dichlorobenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0035	4635	1,2-Dichloroethane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0034	4640	1,1-Dichloroethylene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0043	4645	Cis-1,2-Dichloroethylene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0042	4700	Trans-1,2-Dichloroethylene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0055	4975	Dichloromethane (Methylene Chloride)	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0044	4655	1,2 Dichloropropane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0048	4765	Ethylbenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0053	5100	Styrene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0040	5115	Tetrachloroethylene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0047	5140	Toluene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0036	5160	1,1,1-Trichloroethane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0061	5165	1,1,2-Trichloroethane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0038	5170	Trichloroethylene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0076	5155	1,2,4-Trichlorobenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0032	5235	Vinyl Chloride	2 to 50	±40% fixed acceptance limit				1.2
Drinking Water	0090	5260	Total Xylenes	2 to 50	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
				µg/L					µg/L
Drinking Water	0019	4395	Bromodichloromethane	5 to 50	±20% fixed acceptance limit[14]				4.0
Drinking Water	0018	4400	Bromoform	5 to 50	±20% fixed acceptance limit[14]				4.0
Drinking Water	0020	4575	Chlorodibromomethane	5 to 50	±20% fixed acceptance limit[14]				4.0
Drinking Water	0017	4505	Chloroform	5 to 50	±20% fixed acceptance limit[14]				4.0

NELAC PT for Accreditation
Fields of Proficiency Testing with PTRLs
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			Red = Previous Experimental Analytes	Blue = New Analyte/Header/Footnote				Magenta = Changes	
Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte[1,2]	Conc Range	Acceptance Criteria[3,4,5,6]				NELAC PTRL[7]
					a	b	c	d	
			Volatile Organic Compounds (VOCs)[1] cont'	µg/L					µg/L
Drinking Water	0067	4385	Bromobenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0089	4390	Bromochloromethane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0069	4950	Bromomethane	5 to 50	±40% fixed acceptance limit				3.0
Drinking Water	0079	4435	n-Butylbenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0086	4440	Sec-Butylbenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0085	4445	Tert-Butylbenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0070	4485	Chloroethane	5 to 50	±40% fixed acceptance limit				3.0
Drinking Water	0068	4960	Chloromethane	5 to 50	±40% fixed acceptance limit				3.0
Drinking Water	0071	4535	2-Chlorotoluene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0072	4540	4-Chlorotoluene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0057	4595	Dibromomethane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0066	4615	1,3-Dichlorobenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0088	4625	Dichlorodifluoromethane	5 to 50	±40% fixed acceptance limit				3.0
Drinking Water	0056	4630	1,1-Dichloroethane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0059	4660	1,3-Dichloropropane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0060	4665	2,2-Dichloropropane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0058	4670	1,1-Dichloropropene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0152	4680	Cis-1,3-Dichloropropene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0153	4685	Trans-1,3-Dichloropropene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0081	4835	Hexachlorobutadiene	5 to 50	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				3.0
Drinking Water	0084	4900	Isopropylbenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0083	4910	4-Isopropyltoluene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water		5000	Methyl-tert-butylether (MTBE)	5 to 50	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				3.0
Drinking Water		5005	Naphthalene	5 to 50	± 40% at < 10 ± 30% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0078	5090	n-Propylbenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0063	5105	1,1,1,2-Tetrachloroethane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0065	5110	1,1,2,2-Tetrachloroethane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0077	5150	1,2,3-Trichlorobenzene	5 to 50	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				3.0
Drinking Water	0087	5175	Trichlorofluoromethane	5 to 50	±40% fixed acceptance limit				3.0
Drinking Water	0064	5180	1,2,3-Trichloropropane	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0075	5210	1,2,4-Trimethylbenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
Drinking Water	0082	5215	1,3,5-Trimethylbenzene	2 to 20	± 40% at < 10 ± 20% ≥ 10 fixed acceptance limit				1.2
				µg/L					µg/L
Drinking Water	0045	4570	1,2-Dibromo-3-chloropropane (DBCP)	0.1 to 2	±40% fixed acceptance limit				0.06
Drinking Water	0046	4585	Ethylene Dibromide (EDB)	0.05 to 2	±40% fixed acceptance limit				0.03
Drinking Water		5180	1,2,3-Trichloropropane	0.2 to 2.0	±40% fixed acceptance limit				0.12

NELAC PT for Accreditation

Fields of Proficiency Testing with PTRLs

Drinking Water

Effective January 3, 2012

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Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte[1,2]	Conc Range	Acceptance Criteria[3,4,5,6] a	b	c	d	NELAC PTRL[7]
			Pesticides[1]	µg/L					µg/L
Drinking Water	0093	7005	Alachlor	2 to 20	±45% fixed acceptance limit				1.1
Drinking Water	0256	7025	Aldrin	0.2 to 2.5	0.8618	-0.0012	0.2025	0.0054	0.08
Drinking Water	0094	7065	Atrazine	2 to 20	±45% fixed acceptance limit				1.1
Drinking Water		7160	Butachlor	2 to 20	±45% fixed acceptance limit				1.1
Drinking Water	0097	7250	Chlordane (technical)	2 to 20	±45% fixed acceptance limit				1.1
Drinking Water	0258	7470	Dieldrin	0.5 to 2.5	±45% fixed acceptance limit				0.28
Drinking Water	0011	7540	Endrin	0.2 to 2.5	±30% fixed acceptance limit				0.14
Drinking Water	0095	7685	Heptachlor	0.2 to 2.5	±45% fixed acceptance limit				0.11
Drinking Water	0096	7690	Heptachlor Epoxide (beta)	0.2 to 2.5	±45% fixed acceptance limit				0.11
Drinking Water	0172	6275	Hexachlorobenzene	0.5 to 5	0.8727	0.0048	0.1795	0.0195	0.22
Drinking Water	0112	6285	Hexachlorocyclopentadiene	2 to 20	0.8508	0.0882	0.2716	0.1073	0.49
Drinking Water	0012	7120	Lindane	0.2 to 2.5	±45% fixed acceptance limit				0.11
Drinking Water	0013	7810	Methoxychlor	2 to 20	±45% fixed acceptance limit				1.1
Drinking Water		7835	Metolachlor	2 to 20	±45% fixed acceptance limit				1.1
Drinking Water		7845	Metribuzin	2 to 20	±50% fixed acceptance limit				1.0
Drinking Water	0259	8045	Propachlor	1 to 10	±45% fixed acceptance limit				0.55
Drinking Water	0113	8125	Simazine	2 to 20	±45% fixed acceptance limit				1.1
Drinking Water	0014	8250	Toxaphene (total)	2 to 20	±45% fixed acceptance limit				1.1
Drinking Water	0244	8295	Trifluralin	1 to 10	±45% fixed acceptance limit				0.55
			Carbamates & Vydate	µg/L					µg/L
Drinking Water	0098	7010	Aldicarb	15 to 100	±25% fixed acceptance limit				11
Drinking Water	0099	7015	Aldicarb Sulfone	15 to 100	±25% fixed acceptance limit				11
Drinking Water	0100	7020	Aldicarb Sulfoxide	15 to 80	±25% fixed acceptance limit				11
Drinking Water		7195	Carbaryl	15 to 100	±25% fixed acceptance limit				11
Drinking Water	0101	7205	Carbofuran	15 to 150	±45% fixed acceptance limit				8.3
Drinking Water		7710	3-Hydroxycarbofuran	15 to 80	±20% fixed acceptance limit				12
Drinking Water	0245	7805	Methomyl	15 to 100	±20% fixed acceptance limit				12
Drinking Water	0114	7940	Oxamyl (Vydate)	15 to 100	±25% fixed acceptance limit				11
			Chlorinated Acid Herbicides[13d]	µg/L					µg/L
Drinking Water	0262	8505	Acifluorfen	10 to 100	±50% fixed acceptance limit				5.0
Drinking Water	0015	8545	2,4-D[13e]	10 to 100	±50% fixed acceptance limit				5.0
Drinking Water		8560	2,4-DB	20 to 120	±50% fixed acceptance limit				10
Drinking Water	0115	8555	Dalapon	10 to 100	±50% fixed acceptance limit				5.0
Drinking Water	0247	8595	Dicamba	20 to 100	±50% fixed acceptance limit				10
Drinking Water	0116	8620	Dinoseb	7 to 70	0.8480	0.8414	0.2628	0.0044	3.1
Drinking Water	0102	6605	Pentachlorophenol	1 to 25	±50% fixed acceptance limit				0.50
Drinking Water	0117	8645	Picloram	10 to 100	±50% fixed acceptance limit				5.0
Drinking Water	0016	8650	2,4,5-TP (Silvex)	10 to 100	±50% fixed acceptance limit				5.0
Drinking Water		8655	2,4,5-T	10 to 100	±50% fixed acceptance limit				5.0
			Other Herbicides	µg/L					µg/L
Drinking Water	0137	9390	Diquat[13f]	8 to 40	±50% fixed acceptance limit				4.0
Drinking Water	0138	7525	Endothall[13g]	80 to 500	±50% fixed acceptance limit				40
Drinking Water	0139	9411	Glyphosate	375 to 800	±20% fixed acceptance limit				300

NELAC PT for Accreditation
Fields of Proficiency Testing with PTRLs
Drinking Water
Effective January 3, 2012

Red = Previous Experimental Analytes Blue = New Analyte/Header/Footnote Magenta = Changes

Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte[1,2]	Conc Range	Acceptance Criteria[3,4,5,6] a	b	c	d	NELAC PTRL[7]
			Haloacetic acids	µg/L					µg/L
Drinking Water	0250	9315	Bromochloroacetic Acid	5 to 50	±40% fixed acceptance limit				3.0
Drinking Water	0157	9357	Dibromoacetic Acid	5 to 50	±40% fixed acceptance limit[14]				3.0
Drinking Water	0158	9360	Dichloroacetic Acid	5 to 50	±40% fixed acceptance limit[14]				3.0
Drinking Water	0160	9312	Monobromoacetic Acid	5 to 50	±40% fixed acceptance limit[14]				3.0
Drinking Water	0161	9336	Monochloroacetic Acid	10 to 50	±40% fixed acceptance limit[14]				6.0
Drinking Water	0162	9642	Trichloroacetic Acid	5 to 50	±40% fixed acceptance limit[14]				3.0
			Adipate/Phthalate	µg/L					µg/L
Drinking Water	0134	6062	Di(2-Ethylhexyl) Adipate	8 to 50	0.9817	-0.4239	0.1250	1.4658	2.5
Drinking Water	0136	6065	Di(2-Ethylhexyl) Phthalate	5 to 50	0.9216	1.3142	0.2049	0.7388	2.4
			PCBs in Water[2]	µg/L					µg/L
Drinking Water	0118	9105	PCBs as Decachlorobiphenyl[13h]	0.5 to 5	±100% fixed acceptance limit				0.05
Drinking Water		8872	PCB Aroclor Identification		Correct identification of Aroclor examined				
			PAH	µg/L					µg/L
Drinking Water	0122	5580	Benzo(a)pyrene	0.2 to 2.5	0.8471	-0.0040	0.1854	0.0547	0.02
			Dioxin	pg/L					pg/L
Drinking Water	0252	9618	2,3,7,8-Tetrachloro-dibenzodioxin	20 to 100	0.8642	1.4865	0.1392	1.1445	11

NELAC PT for Accreditation

Fields of Proficiency Testing with PTRLs

Drinking Water

Effective January 3, 2012

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Matrix	EPA	NELAC	Analyte[1,2]	Conc Range	Acceptance Criteria[3,4,5,6]				NELAC PTRL[7]
	Analyte	Analyte			a	b	c	d	
	Code	Code							

1) For volatile and pesticide standards, providers must include a minimum number of analytes using the criteria described below:
PT samples that are to be scored for one to ten analytes must include all of these analytes.
PT sample that are to be scored for ten to twenty analytes must include at least ten of these analytes or 80% ot the total, whichever number is greater.
PT sample that are to be scored for more than twenty analytes must include at least sixteen of these analytes or 60% ot the total, whichever number is greater.
If the calculated percentage of the total number of analytes in the PT sample is a fraction, the fraction shall be rounded up to the next whole number.

2) One sample in every study, containing one Aroclor, selected at random from among the Aroclors listed (1016, 1221, 1232, 1242, 1248, 1254 or 1260) for the analysis of PCBs as decachlorobiphenyl.

3) The acceptance criteria found in 40 CFR Part 141 are incorporated herein by reference. Acceptance criteria for FoPTs not included in 40 CFR Part 141 are presented in this table.
Acceptance limits are set at the Mean ± 2 SD.
Where the a, b, c and d factors are presented, Mean = a*T + b; SD = c*T + d where T is the assigned value.
Where only the c and d factors are presented, Mean = Robust Study Mean; SD = c*X + d where X is the Robust Study Mean.
Where no factors are presented (Study Mean ±3SD), Mean = Robust Study Mean, SD = Robust Study Standard Deviation.
Robust Study Mean and Standard Deviation are generated using statistical analysis of study data set. (ie. Bi-weight, Grubbs, Dixon, etc.)
Quantitative Microbiology acceptance criteria (e.g., HPC) are based on the robust participant Mean and SD determined from each respective PT study, after outlier removal.

4) If the lower acceptance limit generated using the criteria contained in this table is less than (<) 10% of the assigned value, the lower acceptance limits are set at 10% of the assigned value, with the exception of Microbiology analytes.

5) If the lower acceptance limit generated using the criteria contained in this table is greater than (>) 90% of the assigned value, the lower acceptance limits are set at 90% of the assigned value, with the exception of Microbiology analytes.

6) If the upper acceptance limit generated using the criteria contained in this table is less than (<) 110% of the assigned value, the upper acceptance limits are set at 110% of the assigned value, with the exception of Microbiology analytes.

7) NELAC Proficiency Testing Reporting Limits (PTRLs) are provided as guidance to laboratories analyzing NELAC PT samples. These levels are the lowest acceptable results that could be obtained from the lowest spike level for each analyte. The laboratory should report any positive result down to the PTRL. It is recognized that in some cases (especially for analytes that typically exhibit low recovery) the PTRL may be below the standard laboratory reporting limit. However, the laboratory should use a method that is sensitive enough to generate results at the PTRL shown. NELAC PTRLs are also provided as guidance to PT Providers. At a minimum for all analytes with an assigned value equal to "0", the PT Provider should verify that the sample does not contain the analyte at a concentration greater than or equal to the PTRL.

8) The ten-sample set which is provided to the participant laboratories shall contain bacteria that produces the following results when analyzed:
Positive results for total coliforms, fecal coliforms and E.coli.
Positive results for total coliforms and negative results for fecal coliforms and E.coli.
Negative results for total coliforms, fecal coliforms and E.coli.
These limits are for Presence-Absence only.

NELAC PT for Accreditation

Fields of Proficiency Testing with PTRLs

Drinking Water

Effective January 3, 2012

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Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte[1,2]	Conc Range	Acceptance Criteria[3,4,5,6]				NELAC PTRL[7]
					a	b	c	d	

9) The ten-sample set shall be assigned lot numbers and randomly composed of samples as follows:

Two to four samples containing an aerogenic strain of Escherichia which will ensure positive results for total coliforms, fecal coliforms and E.coli.when analyzed by any of the USEPA approved methods.

Two to four samples containing an aerogenic strain of Enterobacter species and/or other microorganism which will ensure positive results for total coliforms and negative result for fecal coliforms and E.coli. when analyzed by any of the USEPA approved methods.

One to two samples containing Pseudomonas species and/or other microorganism which will ensure negative results for total coliforms, fecal coliforms and E.coli. when analyzed by any of the USEPA approved methods.

One to two samples which do not contain any microorganism which ensure negative results for total coliforms, fecal coliforms and E.coli. when analyzed by any of the USEPA approved methods.

10) Laboratories analyzing qualitative sample sets for more than one method in a particular study shall obtain a unique ten-sample set for each method reported as specified in Footnote 9.

11) These limits are for quantitative methods using membrane filtration (MF) or pour-plate (PP) techniques.

12) These limits are for quantitative methods using most probable number (MPN) techniques.

13) The following recommended sample designs, which were used in past USEPA studies, should be used as model designs because other designs may not give equivalent statistics. PT study providers may vary their sample designs from those shown. The specifics within each sample are within the discretion of the PT study Provider.

a) Design criteria for Mercury – 1:1 (mole:mole as Hg) Mercuric Oxide and Methyl Mercuric Chloride.

b) Design criteria for Total Cyanide – uncomplexed, e.g., Potassium Cyanide.

c) Design criterion for Turbidity - Formazin is the source for Turbidity.

d) Design criteria for Chlorinated Acid Herbicides - should be supplied in the acid form of the target herbicide.

e) Design criteria for 2,4-D – should be at least half the butyl ester with the remainder in the acid form.

f) Design criteria for Diquat – Starting material is Diquat Dibromide Monohydrate as required in the method. All assigned values and reported values should be as Diquat.

g) Design criteria for Endothall – Starting material is Endothall Monohydrate as required in the method. All assigned values and reported values should be as Endothall.

h) Design criteria for Decachlorobiphenyl – The source of the Decachlorobiphenyl is one of the following Aroclors: 1016, 1221, 1232, 1242, 1248, 1254, 1260. The assigned value of the Decachlorobiphenyl is to be calculated by the provider from the concentration of the Aroclor used to prepare the sample according to Table 1 of the USEPA Method 508A.

NELAC PT for Accreditation

Fields of Proficiency Testing with PTRLs

Drinking Water

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					a	b	c	d	

14) Laboratories seeking or maintaining NELAP accreditation for Total Trihalomethanes must meet NELAC PT requirements for all 4 Trihalomethane Fields of Proficiency Testing in the given study, by technology/method (Chloroform, Bromoform, Bromodichloromethane, Chlorodibromomethane). Laboratories seeking or maintaining NELAP accreditation for Total Haloacetic Acids must meet NELAC PT requirements for 4 out of 5 regulated Haloacetic Acid Fields of Proficiency Testing in the given PT study, by technology/method (Monochloroacetic Acid, Monobromoacetic Acid, Dichloroacetic Acid, Dibromoacetic Acid, Trichloroacetic Acid).

TNI/NELAP PT for Accreditation

Fields of Proficiency Testing with PTRLs

Non-Potable Water (NPW)

Effective Date: April 1, 2016

Green= Revision 1 Blue = New Analyte Magenta = Changes Red = Revision

Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte	Conc Range	Acceptance Criteria [3,4,5,6]				NELAC PTRL [7]
					a	b	c	d	
			Microbiology	CFU/100 mL					CFU/100 mL
NPW	0233	2500	Total Coliform, MF [8]	20 to 2400	Log transform; ±3 SD				2
NPW	0235	2530	Fecal Coliform, MF [8]	20 to 2400	Log transform; ±3 SD				2
NPW		2525	E.coli, MF [8]	20 to 2400	Log transform; ±3 SD				2
NPW		2520	Enterococci, MF [8]	20 to 1000	Log transform; ±3 SD				2
				MPN/100 mL					MPN/100 mL
NPW	0234	2500	Total Coliform, MPN [9]	20 to 2400	Log transform; ±3 SD				2
NPW	0236	2530	Fecal Coliform, MPN [9]	20 to 2400	Log transform; ±3 SD				2
NPW		2525	E.coli, MPN [9]	20 to 2400	Log transform; ±3 SD				2
NPW		2520	Enterococci, MPN [9]	20 to 1000	Log transform; ±3 SD				2
			Trace Metals	µg/L					µg/L
NPW	0001	1000	Aluminum	200 to 4000	0.9823	9.5889	0.0471	11.2110	144
NPW	0016	1005	Antimony	90 to 900	0.9864	-1.1174	0.0471	6.1230	57
NPW	0002	1010	Arsenic	90 to 900	0.9916	1.2647	0.0422	5.1741	64
NPW	0237	1015	Barium	100 to 2500	±15% fixed acceptance limit				85
NPW	0003	1020	Beryllium	50 to 500	±15% fixed acceptance limit				42
NPW		1025	Boron	800 to 2000	±15% fixed acceptance limit				680
NPW	0004	1030	Cadmium	100 to 1000	±15% fixed acceptance limit				85
NPW	0006	1040	Chromium, total	100 to 1000	±15% fixed acceptance limit				85
NPW	0238	1045	Chromium VI	90 to 900	0.9917	1.0232	0.0476	2.2011	71
NPW	0005	1050	Cobalt	100 to 1000	±15% fixed acceptance limit				85
NPW	0007	1055	Copper	100 to 1000	±15% fixed acceptance limit				85
NPW	0008	1070	Iron	200 to 4000	±15% fixed acceptance limit				170
NPW	0012	1075	Lead	100 to 1500	±15% fixed acceptance limit				85
NPW	0010	1090	Manganese	200 to 2000	±15% fixed acceptance limit				170
NPW	0009	1095	Mercury [10a]	3.0 to 30	±30% fixed acceptance limit				0.9
NPW	0074	1100	Molybdenum	60 to 600	0.9953	-0.1614	0.0372	2.5555	45
NPW	0011	1105	Nickel	200 to 2000	1.0012	1.5795	0.0368	3.8151	168
NPW	0013	1140	Selenium	100 to 1000	±15% fixed acceptance limit				85
NPW	0017	1150	Silver	100 to 1000	±15% fixed acceptance limit				85
NPW	0075	1160	Strontium	50 to 500	±15% fixed acceptance limit				42
NPW	0018	1165	Thallium	80 to 800	0.9932	-0.9634	0.0479	4.2361	54
NPW	0239	1175	Tin	200 to 2000	±30% fixed acceptance limit				140
NPW	0076	1180	Titanium	60 to 300	±15% fixed acceptance limit				51
NPW	0014	1185	Vanadium	50 to 2000	±15% fixed acceptance limit				42
NPW	0015	1190	Zinc	300 to 2000	±15% fixed acceptance limit				255

TNI/NELAP PT for Accreditation
Fields of Proficiency Testing with PTRLs
Non-Potable Water (NPW)
Effective Date: April 1, 2016

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Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte	Conc Range	Acceptance Criteria [3,4,5,6]				NELAC PTRL [7]
					a	b	c	d	
			Demands [10b]	mg/L					mg/L
NPW	0038	1530	5-day BOD [10c]	18 to 230	0.6237	0.7022	0.0928	0.6636	4.9
NPW	0102	1555	Carbonaceous BOD [10c]	18 to 230	0.5648	0.6665	0.0965	0.8253	3.1
NPW	0036	1565	COD [10d]	30 to 250	0.9843	-0.3171	0.0432	3.0191	16
NPW	0037	2040	TOC [10e]	6.0 to 100	0.9926	0.1680	0.0473	0.3536	4.2
			Minerals	mg/L					mg/L
NPW	0027	1505	Alkalinity, total ($CaCO_3$)	25 to 400	±20% at < 40; ±15% at ≥ 40 fixed acceptance limit				20
NPW		1540	Bromide	1.0 to 10	1.0098	-0.0533	0.0400	0.0912	0.56
NPW	0023	1035	Calcium	10 to 100	±15% fixed acceptance limit				8.5
NPW	0028	1575	Chloride	35 to 275	1.0005	0.0490	0.0376	0.3716	30
NPW	0029	1730	Fluoride	0.4 to 4	0.9748	0.0156	0.0487	0.0277	0.26
NPW		1550	Calcium hardness as CaCO3	25 to 250	±15% fixed acceptance limit				21
NPW	0022	1755	Hardness, total ($CaCO_3$)	40 to 415	±15% fixed acceptance limit				34
NPW	0024	1085	Magnesium	4.0 to 40	±15% fixed acceptance limit				3.4
NPW	0026	1125	Potassium	4.0 to 40	±20% fixed acceptance limit				3.2
NPW	0025	1155	Sodium	10 to 100	±20% fixed acceptance limit				8.0
NPW	0020	1610	Spec. Cond. (25°C)	200 to 1200 µmhos/cm	±10% fixed acceptance limit				180 µmhos/cm
NPW	0030	2000	Sulfate	5.0 to 125	0.9880	-0.2130	0.0473	0.3309	3.0
NPW		2005	Sulfide	2.0 to 10	0.9657	-0.1271	0.1205	0.2816	0.20
NPW	0021	1955	Total Dissolved Solids at 180°C	140 to 800	1.0000	0.0000	0.0000	15.0000	95
NPW	0105	1950	Total Solids	140 to 800	1.0000	0.0000	0.0000	15.0000	95
			Nutrients	mg/L					mg/L
NPW	0031	1515	Ammonia as N	1.0 to 20	0.9923	0.0567	0.0583	0.0914	0.60
NPW	0032	1810	Nitrate as N	2.0 to 25	0.9975	-0.0005	0.0506	0.0642	1.50
NPW		1820	Nitrate-nitrite as N	2.5 to 25	0.9957	-0.0010	0.0509	0.0400	1.99
NPW		1840	Nitrite as N	0.4 to 4.0	1.0017	-0.0030	0.0377	0.0250	0.28
NPW	0033	1870	Orthophosphate as P	0.5 to 5.5	±15% fixed acceptance limit				0.42
NPW	0034	1795	Total Kjeldahl-Nitrogen [10f]	3.0 to 35	0.9701	0.2283	0.0680	0.1906	1.95
NPW	0035	1910	Total Phosphorus	0.5 to 10	0.9932	0.0084	0.0506	0.0254	0.35

TNI/NELAP PT for Accreditation
Fields of Proficiency Testing with PTRLs
Non-Potable Water (NPW)
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Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte	Conc Range	Acceptance Criteria [3,4,5,6]				NELAC PTRL [7]
					a	b	c	d	
			Misc. Analytes	mg/L					mg/L
NPW		1500	Acidity, as CaCO3	650 to 1800	±10% fixed acceptance limit				585
NPW		1605	Color	10 to 75 PC units	0.9474	0.6098	0.0367	2.4407	1.7 PC units
NPW	0072	1960	Total Suspended Solids	20 to 100	0.9728	-0.6338	0.0300	1.5793	12
NPW	0019	1900	pH [10g]	5.0 to 10 units	± 0.2 units fixed acceptance limit				Not applicable
NPW	0071	1645	Total Cyanide [10h]	0.1 to 1	±35% fixed acceptance limit				0.065
NPW	0097	1905	Total Phenolics (4AAP) [10i]	0.5 to 5	0.6408	0.0250	0.1038	0.0082	0.16
NPW	0098	1940	Total Residual Chlorine	0.5 to 3.0	0.9345	0.0392	0.0688	0.0073	0.38
NPW		1965	Settleable solids	5.0 to 50 mL/L	1.0436	-0.0108	0.0597	0.4546	2.9 mL/L
NPW		1990	Silica as SiO2	50 to 250	±25% fixed acceptance limit				38
NPW		2025	Surfactants - MBAS	0.2 to 1.0	1.0421	-0.0068	0.1326	0.0046	0.10
NPW		2055	Turbidity [10j]	2.0 to 30 NTU	1.0040	-0.0368	0.0475	0.1575	1.2 NTU
NPW		1970	Volatile solids, Total	100 to 500	0.9644	-4.7559	0.0182	14.9450	41
			Low Level Analytes [11]						
NPW		1095	Mercury [10a]	20 to 100 ng/L	0.9910	0.2064	0.0432	2.5774	9.7
NPW		1940	Total Residual Chlorine	50 to 250 µg/L	1.0000	0.0000	0.0000	20.0000	5.0

TNI/NELAP PT for Accreditation
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Non-Potable Water (NPW)
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Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte	Conc Range	Acceptance Criteria [3,4,5,6]				NELAC PTRL [7]
					a	b	c	d	
			Volatile Aromatics [1]	µg/L					µg/L
NPW	0065	4375	Benzene	10 to 120	±30% fixed acceptance limit				7.0
NPW	0094	4610	1,2-Dichlorobenzene	10 to 120	±30% fixed acceptance limit				7.0
NPW	0096	4615	1,3-Dichlorobenzene	10 to 120	±30% fixed acceptance limit				7.0
NPW	0095	4620	1,4-Dichlorobenzene	10 to 120	±30% fixed acceptance limit				7.0
NPW	0066	4765	Ethylbenzene	10 to 120	±30% fixed acceptance limit				7.0
NPW	0222	5005	Naphthalene	15 to 150	0.8785	1.4343	0.1335	0.7561	6.3
NPW		5100	Styrene	20 to 120	±35% fixed acceptance limit				13.0
NPW	0067	5140	Toluene	10 to 120	±30% fixed acceptance limit				7.0
NPW	0092	5155	1,2,4-Trichlorobenzene	15 to 150	0.9160	-1.3028	0.1473	0.5100	4.3
NPW		5210	1,2,4-Trimethylbenzene	10 to 120	±35% fixed acceptance limit				6.5
NPW		5215	1,3,5-Trimethylbenzene	10 to 120	±35% fixed acceptance limit				6.5
NPW		5240	m/p-Xylenes	10 to 150	±40% fixed acceptance limit				6.0
NPW		5250	o-Xylene	10 to 150	±40% fixed acceptance limit				6.0
NPW	0242	5260	Xylenes, total [12]	20 to 300	±40% fixed acceptance limit				12
			Volatile Ketones/Ethers [1]	µg/L					µg/L
NPW		4315	Acetone	20 to 200	0.8856	3.5838	0.2028	1.7474	3.9
NPW		4860	2-Hexanone	20 to 200	1.0054	-1.1748	0.1534	1.7764	4.4
NPW		4995	4-Methyl-2-pentanone (MIBK)	20 to 200	1.0022	-1.0337	0.0934	4.1819	2.0
NPW		5000	Methyl tert-butyl ether (MTBE)	15 to 150	1.0233	-0.3620	0.1112	0.3083	9.0

TNI/NELAP PT for Accreditation

Fields of Proficiency Testing with PTRLs

Non-Potable Water (NPW)

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					a	b	c	d	
			Volatile Halocarbons [1]	µg/L					µg/L
NPW	0060	4395	Bromodichloromethane	10 to 100	±40% fixed acceptance limit				6.0
NPW	0062	4400	Bromoform	10 to 100	±40% fixed acceptance limit				6.0
NPW	0243	4950	Bromomethane	20 to 120	± 60% fixed acceptance limit				8.0
NPW	0058	4455	Carbon tetrachloride	15 to 150	0.9577	0.0612	0.1269	0.3443	7.7
NPW	0064	4475	Chlorobenzene	10 to 120	±30% fixed acceptance limit				7.0
NPW	0244	4485	Chloroethane	20 to 120	± 60% fixed acceptance limit				8.0
NPW	0055	4505	Chloroform	10 to 100	±30% fixed acceptance limit				7.0
NPW	0245	4960	Chloromethane	20 to 120	± 60% fixed acceptance limit				8.0
NPW	0061	4575	Dibromochloromethane	10 to 100	±40% fixed acceptance limit				6.0
NPW		4570	1,2-Dibromo-3-chloropropane (DBCP)	15 to 150	±40% fixed acceptance limit				9.0
NPW		4585	1,2-Dibromoethane (EDB)	10 to 120	±35% fixed acceptance limit				6.5
NPW		4595	Dibromomethane	10 to 120	±35% fixed acceptance limit				6.5
NPW		4630	1,1-Dichloroethane	10 to 150	0.9977	0.2117	0.1227	0.0174	6.4
NPW	0054	4635	1,2 Dichloroethane	15 to 150	0.9843	1.3728	0.0912	0.4693	10.6
NPW	0246	4640	1,1-Dichloroethene	10 to 150	1.0034	0.6630	0.1447	0.0521	6.2
NPW		4645	cis-1,2-Dichloroethene	10 to 150	0.9973	0.3699	0.1095	0.0036	7.0
NPW	0247	4700	trans-1,2-Dichloroethene	10 to 120	±40% fixed acceptance limit				6.0
NPW	0248	4655	1,2-Dichloropropane	10 to 150	±30% fixed acceptance limit				7.0
NPW		4680	cis-1,3-Dichloropropene	10 to 120	±35% fixed acceptance limit				6.5
NPW	0249	4685	trans-1,3-Dichloropropene	10 to 120	±35% fixed acceptance limit				6.5
NPW	0063	4975	Methylene Chloride	10 to 120	±40% fixed acceptance limit				6.0
NPW		5105	1,1,1,2-Tetrachloroethane	15 to 150	±35% fixed acceptance limit				9.8
NPW	0250	5110	1,1,2,2-Tetrachloroethane	15 to 150	±35% fixed acceptance limit				9.8
NPW	0059	5115	Tetrachloroethene	10 to 150	0.9416	-0.5063	0.1189	0.3441	4.3
NPW	0056	5160	1,1,1-Trichloroethane	10 to 100	±40% fixed acceptance limit				6.0
NPW	0251	5165	1,1,2-Trichloroethane	15 to 150	±30% fixed acceptance limit				10.5
NPW	0057	5170	Trichloroethene	10 to 100	0.9611	0.5720	0.1077	0.2478	6.2
NPW	0252	5175	Trichlorofluoromethane	20 to 120	± 60% fixed acceptance limit				8.0
NPW		5180	1,2,3-Trichloropropane	15 to 150	0.9867	-0.4721	0.1630	0.9605	4.1
NPW	0253	5235	Vinyl chloride	20 to 120	± 60% fixed acceptance limit				8.0
			Low-Level Halocarbons [1, 11]	µg/L					µg/L
NPW		4570	1,2-Dibromo-3-chloropropane (DBCP)	0.2 to 2.0	0.9542	0.0359	0.1200	0.0161	0.11
NPW		4585	1,2-Dibromoethane (EDB)	0.2 to 2.0	0.9341	0.0293	0.1090	0.0239	0.08
NPW		5180	1,2,3-Trichloropropane	0.2 to 2.0	0.9284	0.0534	0.1257	0.0117	0.13

TNI/NELAP PT for Accreditation

Fields of Proficiency Testing with PTRLs

Non-Potable Water (NPW)

Effective Date: April 1, 2016

Green= Revision 1 | Blue = New Analyte | Magenta = Changes | Red = Revision

Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte	Conc Range	Acceptance Criteria [3,4,5,6]				NELAC PTRL [7]
					a	b	c	d	
			Volatile Petroleum Hydrocarbons	µg/L					µg/L
NPW		9408	Gasoline range organics (GRO) [13]	400 to 4000	1.0683	-7.7234	0.2162	35.0439	55
			Base/Neutrals [1]	µg/L					µg/L
NPW	0189	5500	Acenaphthene	10 to 200	0.7748	0.8506	0.1427	0.1159	4.0
NPW	0190	5505	Acenaphthylene	10 to 200	0.8029	-0.2974	0.1485	0.1111	2.9
NPW	0192	5555	Anthracene	10 to 200	0.7986	1.7870	0.1229	0.7303	3.9
NPW	0177	5575	Benzo(a)anthracene	10 to 200	0.8381	0.5699	0.1162	0.6075	3.6
NPW	0254	5670	Benzyl butyl phthalate	50 to 200	0.8496	-2.1863	0.1776	0.0752	13.4
NPW	0178	5585	Benzo(b)fluoranthene	20 to 200	0.8327	0.1531	0.1497	0.1078	7.5
NPW	0179	5600	Benzo(k)fluoranthene	20 to 200	0.8223	1.996	0.1862	1.126	7.7
NPW	0180	5590	Benzo(g,h,i)perylene	10 to 200	0.8261	1.5562	0.1556	0.0166	5.1
NPW	0255	5580	Benzo(a)pyrene	10 to 200	0.8207	-0.0550	0.1484	0.4349	2.4
NPW	0198	5660	4-Bromophenyl-phenylether	20 to 200	0.8081	3.0645	0.1325	0.8996	8.6
NPW	0195	5760	bis(2-Chloroethoxy)methane	20 to 200	0.7615	0.4890	0.1193	1.5633	3.9
NPW	0196	5765	bis(2-Chloroethyl)ether	20 to 200	0.7090	2.3607	0.1529	0.4801	5.9
NPW	0197	4659	2,2'-Oxybis(1-Chloropropane)[18]	30 to 200	0.7285	1.6917	0.1303	2.9025	3.1
NPW	0256	6065	Bis(2-ethylhexyl) phthalate	20 to 200	0.8065	2.5761	0.1474	1.6124	5.0
NPW	0204	5825	4-Chlorophenyl-phenylether	20 to 200	0.7669	3.7466	0.1417	0.2303	9.9
NPW	0203	5795	2-Chloronaphthalene	20 to 200	0.7102	2.4854	0.1477	0.5079	6.3
NPW	0181	5855	Chrysene	10 to 200	0.8180	2.3274	0.1351	0.2137	5.8
NPW	0182	5895	Dibenzo(a,h)anthracene	20 to 200	0.8079	2.3890	0.1497	0.8729	6.9
NPW		5905	Dibenzofuran	30 to 200	0.7411	2.7181	0.1159	1.0735	11.3
NPW		4610	1,2-Dichlorobenzene	20 to 200	0.6365	0.7906	0.1517	2.2155	2.0
NPW		4615	1,3-Dichlorobenzene	20 to 200	0.5921	3.0260	0.1787	0.3464	3.1
NPW		4620	1,4-Dichlorobenzene	20 to 200	0.5671	3.6005	0.1640	0.4826	3.7
NPW	0185	5945	3,3'-Dichlorobenzidine	50 to 200	0.901	-0.5596	0.199	2.5071	5.0
NPW	0208	6070	Diethyl phthalate	50 to 200	0.7492	3.3637	0.1805	2.0213	8.9
NPW	0209	6135	Dimethyl phthalate	50 to 200	0.6375	3.9631	0.2524	0.8174	11.5
NPW	0205	5925	Di-n-butyl phthalate	40 to 200	0.7797	5.1233	0.1490	0.8776	15.8
NPW	0186	6185	2,4-Dinitrotoluene	20 to 200	0.8219	0.4137	0.1183	1.7449	4.5
NPW	0210	6190	2,6-Dinitrotoluene	20 to 200	0.7999	0.4770	0.1316	0.1368	8.2
NPW	0211	6200	Di-n-octyl phthalate	30 to 200	0.8186	2.8779	0.1724	1.2382	8.2
NPW	0212	6265	Fluoranthene	30 to 200	0.8087	2.9863	0.1272	0.0642	15.6
NPW	0213	6270	Fluorene	10 to 200	0.7619	3.7583	0.1165	1.0349	4.8
NPW	0214	6275	Hexachlorobenzene	20 to 200	0.8202	0.2263	0.1238	0.1297	8.8
NPW	0215	4835	Hexachlorobutadiene	50 to 200	0.6286	2.6591	0.1616	1.9082	4.3
NPW	0216	6285	Hexachlorocyclopentadiene	50 to 200	0.6216	-4.4226	0.2049	4.3222	5.0
NPW	0217	4840	Hexachloroethane	50 to 200	0.5921	-0.0657	0.1640	0.5308	3.3
NPW	0218	6315	Indeno(1,2,3, cd)pyrene	30 to 200	0.7115	5.0289	0.1430	1.4299	9.2
NPW	0219	6320	Isophorone	20 to 200	0.7981	0.7053	0.1437	0.3000	7.1
NPW		6385	2-Methylnaphthalene	20 to 200	0.6983	2.0844	0.1361	2.1436	2.0

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Fields of Proficiency Testing with PTRLs

Non-Potable Water (NPW)

Effective Date: April 1, 2016

Green= Revision 1 Blue = New Analyte Magenta = Changes Red = Revision

Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte	Conc Range	Acceptance Criteria [3,4,5,6]				NELAC PTRL [7]
					a	b	c	d	
			Base/Neutrals [1] cont'	µg/L					µg/L
NPW	0222	5005	Naphthalene	20 to 200	0.6749	3.5514	0.1441	1.2975	4.5
NPW	0226	5015	Nitrobenzene	20 to 200	0.7463	0.9864	0.1388	0.4589	6.2
NPW	0227	6530	N-Nitrosodimethylamine	75 to 200	0.4665	7.3433	0.1652	3.9997	7.5
NPW	0230	6545	N-Nitroso-di-n-propylamine	30 to 200	0.7913	-0.0510	0.1541	0.1328	9.4
NPW	0229	6535	N-Nitrosodiphenylamine	30 to 200	0.7740	0.6711	0.2016	0.0494	5.6
NPW	0231	6615	Phenanthrene	10 to 200	0.8001	2.8698	0.1110	0.9485	4.7
NPW	0187	6665	Pyrene	10 to 200	0.8476	1.0097	0.1490	0.0530	4.9
NPW	0092	5155	1,2,4-Trichlorobenzene	20 to 200	0.6769	1.1166	0.1493	1.8128	2.0
			Acids [1]	µg/L					µg/L
NPW	0161	5700	4-Chloro-3-methylphenol	30 to 200	0.7998	0.6264	0.1421	0.0397	11.7
NPW	0162	5800	2-Chlorophenol	30 to 200	0.7292	1.4640	0.1518	0.0174	9.6
NPW	0163	6000	2,4-Dichlorophenol	30 to 200	0.7362	2.8458	0.1433	0.0585	11.9
NPW		6005	2,6-Dichlorophenol	30 to 200	0.7512	3.7563	0.1564	0.0312	12.1
NPW	0165	6130	2,4-Dimethylphenol	40 to 200	0.7496	1.4509	0.1601	0.0953	11.9
NPW	0167	6175	2,4-Dinitrophenol	100 to 200	0.6531	3.5920	0.1695	8.5727	10
NPW	0168	6360	2-Methyl-4,6-Dinitrophenol	40 to 200	0.8108	3.6290	0.1573	2.1683	10.7
NPW		6400	2-Methylphenol (o-Cresol)	40 to 200	0.6821	2.2126	0.1529	0.5485	9.5
NPW		6410	4-Methylphenol (p-Cresol) [14]	50 to 200	0.6531	2.1854	0.2008	0.7807	5.0
NPW	0171	6490	2-Nitrophenol	50 to 200	0.7631	1.1486	0.1272	2.4547	12.9
NPW	0173	6500	4-Nitrophenol	100 to 200	0.5591	-1.0075	0.2511	1.9409	10
NPW	0174	6625	Phenol	100 to 200	0.557	0.5929	0.253	1.0269	10
NPW	0158	6605	Pentachlorophenol	40 to 200	0.8469	-0.7338	0.1561	1.5178	9.9
NPW	0175	6835	2,4,5-Trichlorophenol	30 to 200	0.7726	3.2199	0.1362	0.9916	11.2
NPW	0159	6840	2,4,6-Trichlorophenol	30 to 200	0.7880	0.8051	0.1406	0.0280	11.7

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Fields of Proficiency Testing with PTRLs

Non-Potable Water (NPW)

Effective Date: April 1, 2016

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Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte	Conc Range	Acceptance Criteria [3,4,5,6]				NELAC PTRL [7]
					a	b	c	d	
			PCBs in Water [2]	µg/L					µg/L
NPW	0040	8880	Aroclor 1016	2.0 to 10	0.8318	0.1991	0.1591	0.0384	0.8
NPW	0041	8885	Aroclor 1221	2.0 to 10	0.8318	0.1991	0.1591	0.0384	0.8
NPW	0042	8890	Aroclor 1232	2.0 to 10	0.8318	0.1991	0.1591	0.0384	0.8
NPW	0040	8895	Aroclor 1242	2.0 to 10	0.8318	0.1991	0.1591	0.0384	0.8
NPW	0044	8900	Aroclor 1248	2.0 to 10	0.8318	0.1991	0.1591	0.0384	0.8
NPW	0045	8905	Aroclor 1254	2.0 to 10	0.8318	0.1991	0.1591	0.0384	0.8
NPW	0046	8910	Aroclor 1260	2.0 to 10	0.8318	0.1991	0.1591	0.0384	0.8
			Organochlorine Pesticides [1]	µg/L					µg/L
NPW	0047	7025	Aldrin	1.0 to 15	0.8524	-0.0159	0.1655	0.0002	0.34
NPW	0079	7110	alpha-BHC	2.0 to 20	0.8996	0.0151	0.1505	0.0349	0.81
NPW	0080	7115	beta-BHC	2.0 to 20	0.8889	0.1961	0.1372	0.0777	0.92
NPW	0081	7105	delta-BHC	2.0 to 20	0.9031	0.1036	0.1525	0.0673	0.79
NPW	0082	7120	gamma-BHC (Lindane)	2.0 to 20	0.8959	0.1095	0.1528	0.0189	0.93
NPW		7240	alpha-Chlordane	1.0 to 10	0.8842	0.0542	0.1423	0.0348	0.41
NPW		7245	gamma-Chlordane	1.0 to 10	0.8617	0.1041	0.1323	0.0716	0.35
NPW	0053	7250	Chlordane (total)	3.0 to 25	0.8501	0.4121	0.1540	0.0381	1.46
NPW	0049	7355	4,4'-DDD	2.0 to 10	0.9271	0.03839	0.1227	0.1763	0.63
NPW	0050	7360	4,4'-DDE	1.0 to 10	0.8793	0.0718	0.1468	0.0395	0.39
NPW	0051	7365	4,4'-DDT	1.0 to 10	0.8987	0.1076	0.1680	0.0337	0.40
NPW	0048	7470	Dieldrin	1.0 to 15	0.9126	0.0323	0.1327	0.0240	0.47
NPW	0083	7510	Endosulfan I	4.0 to 20	0.8698	-0.0604	0.1548	0.0549	1.40
NPW	0084	7515	Endosulfan II	4.0 to 20	0.8765	0.0994	0.1490	0.0912	1.54
NPW	0085	7520	Endosulfan sulfate	4.0 to 20	0.8752	0.5312	0.1348	0.2091	1.79
NPW	0086	7540	Endrin	2.0 to 20	0.9183	0.0706	0.1594	0.0277	0.87
NPW	0087	7530	Endrin aldehyde	4.0 to 20	0.8585	0.4845	0.1571	0.2054	1.42
NPW		7535	Endrin ketone	4.0 to 20	0.8951	0.3702	0.1135	0.1902	2.0
NPW	0052	7685	Heptachlor	1.0 to 10	0.8470	0.0457	0.1596	0.0402	0.29
NPW	0078	7690	Heptachlor Epoxide (beta)	1.0 to 10	0.9176	0.0041	0.1342	0.0268	0.44
NPW	0234	7810	Methoxychlor	2.0 to 20	0.9115	0.2801	0.1467	0.2290	0.54
NPW	0241	8250	Toxaphene	20 to 100	0.8087	1.8908	0.1991	0.5080	4.59

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Fields of Proficiency Testing with PTRLs
Non-Potable Water (NPW)
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Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte	Conc Range	Acceptance Criteria [3,4,5,6]				NELAC PTRL [7]
					a	b	c	d	
			Herbicides [1]	µg/L					µg/L
NPW	0257	8545	2,4-D	2 to 10	0.7204	0.2995	0.2543	0.0297	0.20
NPW	0258	8595	Dicamba	2 to 10	0.7848	0.2788	0.1754	0.1455	0.40
NPW	0140	8655	2,4,5-T	2 to 10	0.8132	0.1393	0.1850	0.1353	0.20
NPW	0259	8650	2,4,5-TP (Silvex)	2 to 10	0.8349	0.1516	0.2046	0.0195	0.50
			Low Level PAHs [1]	µg/L					µg/L
NPW		5500	Acenaphthene	2.0 to 20	0.7600	0.1476	0.1456	0.0021	0.79
NPW		5505	Acenaphthylene	2.0 to 20	0.7856	0.0418	0.1133	0.0687	0.73
NPW		5555	Anthracene	0.5 to 5.0	0.8151	0.0194	0.1714	0.0115	0.14
NPW		5575	Benzo(a)anthracene	0.5 to 5.0	0.9012	-0.0236	0.0614	0.0462	0.20
NPW		5580	Benzo(a)pyrene	0.5 to 5.0	0.7745	0.0824	0.1162	0.0270	0.21
NPW		5585	Benzo(b)fluoranthene	0.5 to 5.0	0.8217	0.0544	0.1167	0.0144	0.25
NPW		5590	Benzo(g,h,i)perylene	0.5 to 5.0	0.7683	0.0737	0.1641	0.0088	0.18
NPW		5600	Benzo(k)fluoranthene	0.5 to 5.0	0.8943	-0.0069	0.1245	0.0108	0.22
NPW		5855	Chrysene	0.5 to 5.0	0.8883	0.0132	0.1046	0.0235	0.23
NPW		5895	Dibenz(a,h)anthracene	0.5 to 5.0	0.7914	0.0640	0.1377	0.0520	0.10
NPW		6265	Fluoranthene	0.5 to 5.0	0.8565	0.0211	0.1064	0.0128	0.25
NPW		6270	Fluorene	2.0 to 10	0.7863	0.0472	0.1153	0.0631	0.74
NPW		6315	Indeno(1,2,3-cd)pyrene	0.5 to 5.0	0.8224	0.0623	0.1316	0.0267	0.20
NPW		5005	Naphthalene	2.0 to 10	0.7279	0.0977	0.1251	0.0803	0.56
NPW		6615	Phenanthrene	0.5 to 5.0	0.8332	0.0256	0.1099	0.0118	0.24
NPW		6665	Pyrene	0.5 to 5.0	0.8468	0.0435	0.1023	0.0095	0.28
			Petroleum Hydrocarbons						
NPW		9369	Diesel range organics (DRO) [15]	800 to 6000 µg/L	0.7790	-96.0467	0.1386	109.1897	80 µg/L
NPW	0104	1803	n-Hexane Extractable Material (O&G) [10k,16]	20 to 200 mg/L	0.9400	-0.4116	0.0545	2.0789	8.8 mg/L
NPW		1935	non-Polar Extractable Material (TPH) [17]	20 to 200 mg/L	0.9692	-1.1573	0.1586	0.3709	7.6 mg/L

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Fields of Proficiency Testing with PTRLs

Non-Potable Water (NPW)

Effective Date: April 1, 2016

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Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte	Conc Range	Acceptance Criteria [3,4,5,6]				NELAC PTRL [7]
					a	b	c	d	

1) For volatiles, base/neutrals, acids, organochlorine pesticides, herbicides, and low level PAHs, providers must include a minimum number of analytes using the criteria described below:

PT samples that are to be scored for one to ten analytes must include all of these analytes.

PT samples that are to be scored for ten to twenty analytes must include at least ten of these analytes or 80% ot the total, whichever number is greater.

PT samples that are to be scored for more than twenty analytes must include at least sixteen of these analytes or 60% ot the total, whichever number is greater.

If the calculated percentage of the total number of analytes in the PT sample is a fraction, the fraction shall be rounded up to the next whole number.

2) One sample (minimum) in every study, containing one Aroclor, selected at random from among the Aroclors listed above.

3) Acceptance limits are set at the Mean ± 3 SD

Where the a, b, c and d factors are presented, Mean = a*T + b; SD = c*T + d where T is the assigned value.

Where only the c and d factors are presented, Mean = Robust Study Mean; SD = c*X + d where X is the Robust Study Mean.

Where no factors are presented (Study Mean ±3SD), Mean = Robust Study Mean, SD = Robust Study Standard Deviation.

Robust Study Mean and Standard Deviation are generated using statistical analysis of study data set. (ie. Bi-weight, Grubbs, Dixon, etc.)

Quantitative Microbiology acceptance criteria are based on the robust participant Mean and SD determined from each respective PT study

4) If the lower acceptance limit generated using the criteria contained in this table is less than (<) 10% of the assigned value, the lower acceptance limits are set at 10% of the assigned value with the exception of microbiology analytes.

5) If the lower acceptance limit generated using the criteria contained in this table is greater than 90% of the assigned value, the lower acceptance limits are set at 90% of the assigned value with the exception of microbiology analytes.

6) If the upper acceptance limit generated using the criteria contained in this table is less than 110% of the assigned value, the upper acceptance limits are set at 110% of the assigned value with the exception of microbiology analytes.

7) NELAC Proficiency Testing Reporting Limits (PTRLs) are provided as guidance to laboratories analyzing NELAC PT samples. These levels are the lowest acceptable results that could be obtained from the lowest spike level for each analyte. The laboratory should report any positive result down to the PTRL. It is recognized that in some cases (especially for analytes that typically exhibit low recovery) the PTRL may be below the standard laboratory reporting limit. However, the laboratory should use a method that is sensitive enough to generate results at the PTRL shown. NELAC PTRLs are also provided as guidance to PT Providers. At a minimum for all analytes with an assigned value equal to "0", the PT Provider should verify that the sample does not contain the analyte at a concentration greater than or equal to the PTRL.

8) These limits are for quantitative methods using membrane filtration techniques.

9) These limits are for quantitative methods using most probable number techniques.

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					a	b	c	d	

10) The following recommended sample designs, which were used in past USEPA studies, should be used as model designs because other designs may not give equivalent statistics. PT study providers may vary their sample designs from those shown. The specifics within each sample are within the discretion of the PT study Provider.

a) Design criterion for Mercury – 1:1 (mole:mole as Hg) Mercuric Oxide and Methyl Mercuric Chloride.

b) Design criteria for Demands – 1:1 Glucose and Glutamic Acid.

c) Design criteria for 5-Day BOD and Carbonaceous BOD – The assigned value used for BOD and CBOD is derived from the linear relationship between the BOD or CBOD value and the concentration of Glucose-Glutamic Acid (GGA) or Potassium Hydrogen Phthalate (KHP) used for the formulation. For example, 150 mg/L each of Glucose & of Glutamic Acid produces a BOD of 198 mg/L, and 300 mg/L KHP produces a BOD of 240 mg/L. 0 mg/L GGA or KHP would produce a BOD value of 0 mg/L.

d) Design criterion for Chemical Oxygen Demand – The assigned value of COD is (1.066 times mg Glucose plus 0.9787 times mg Glutamic Acid) divided by total liters of sample adjusted for required dilutions.

e) Design criterion for Total Organic Carbon – The assigned value of TOC is (0.4000 times mg Glucose plus 0.4082 times mg Glutamic Acid) divided by total liters of sample adjusted for required dilutions.

f) Design criterion for Total Kjeldahl Nitrogen – Glycine is the source of TKN.

g) Design criterion for pH – in separate solution (use buffer formulation from the CRC chemical handbook).

h) Design criterion for Total Cyanide – Potassium Ferricyanide.

i) Design criterion for Total Phenolics (4AAP) – 40% Phenol, 20% 2-Chlorophenol, 20% 2,4-Dinitrophenol, 20% 2,4-Dichlorophenol (mole %), calculated as mg/L Phenol.

j) Design criterion for Turbidity - Formazin is the source for Turbidity.

k) Design criterion for Oil and Grease – 1:1 Paraffin oil and cooking oil, vacuum pump oil, or similar mixture that does not contain volatile organics.

11) The Low Level Analytes' concentration ranges and acceptance criteria are specifically intended for technologies/methods that can achieve the listed PTRL.

12) Volatiles Aromatics must contain all three Xylene isomers. The concentration range of o-Xylene and m&p-Xylene is 10-150 µg/L each.

13) Gasoline Range Organics (GRO) per purge-and-trap extraction followed by chromatographic analysis. GRO is defined as the carbon range between n-C5 and n-C10.

TNI/NELAP PT for Accreditation

Fields of Proficiency Testing with PTRLs

Non-Potable Water (NPW)

Effective Date: April 1, 2016

Green= Revision 1 | Blue = New Analyte | Magenta = Changes | Red = Revision

Matrix	EPA Analyte Code	NELAC Analyte Code	Analyte	Conc Range	Acceptance Criteria [3,4,5,6]				NELAC PTRL [7]
					a	b	c	d	

14) Laboratories seeking or maintaining NELAP accreditation for Non-Potable Water 4-Methylphenol or the coeluting isomer pair of 3-Methylphenol and 4-Methylphenol must meet the NELAC PT requirements for this Field of Proficiency Testing (4-Methylphenol).

15) Diesel Range Organics (DRO) per solvent extraction followed by chromatographic analysis. DRO is defined as the carbon range between n-C_{10} and n-C_{28}.

16) n-Hexane Extractable Material (HEM) per solvent extraction followed by gravimetric or infrared spectrometric analysis (Oil & Grease).

17) non-Polar Extractable Material per solvent extraction and Silica Gel Treated (SGT) followed by gravimetric or infrared spectrometric analysis (Total Petroleum Hydrocarbons).

18) Also known as Bis(2-chloro-1-methylethyl) Ether; formerly inaccurately labeled as Bis(2-chloroisopropyl) Ether.

ERA
A Waters Company

Reference Materials

▪ Certificate of Analysis ▪

Product: WatR™ Pollution Volatiles
Catalog Number: 710
Lot No. P245-710
Certificate Issue Date: October 28, 2015
Expiration Date: May 06, 2018
Revision Number: Original

CERTIFICATION

Parameter	Certified Value[1]	Uncertainty[2]	QC Performance Acceptance Limits[3]	PT Performance Acceptance Limits[4]
	µg/L	%	µg/L	µg/L
Acetone	<3.90	-	-	-
Acetonitrile	<5.00	-	-	-
Acrolein	<5.00	-	-	-
Acrylonitrile	<5.00	-	-	-
Benzene	68.2	3.34	53.2 - 81.2	47.7 - 88.7
Bromobenzene	<5.00	-	-	-
Bromochloromethane	<5.00	-	-	-
Bromodichloromethane	<6.00	-	-	-
Bromoform	<6.00	-	-	-
Bromomethane	<8.00	-	-	-
2-Butanone (MEK)	<5.00	-	-	-
n-Butylbenzene	<5.00	-	-	-
sec-Butylbenzene	<5.00	-	-	-
tert-Butylbenzene	<5.00	-	-	-
tert-Butyl methyl ether (MTBE)	89.4	6.34	70.4 - 114	60.4 - 122
Carbon disulfide	<5.00	-	-	-
Carbon tetrachloride	16.9	4.27	11.0 - 21.1	8.78 - 23.7
Chlorobenzene	94.1	4.60	74.5 - 112	65.9 - 122
Chlorodibromomethane	<6.00	-	-	-
Chloroethane	<8.00	-	-	-
2-Chloroethylvinylether	<5.00	-	-	-
Chloroform	34.3	1.77	27.0 - 41.8	24.0 - 44.6
Chloromethane	<8.00	-	-	-
2-Chlorotoluene	<5.00	-	-	-

ACCREDITED ACCREDITED

Parameter	Certified Value[1]	Uncertainty[2]	QC Performance Acceptance Limits[3]	PT Performance Acceptance Limits[4]
	µg/L	%	µg/L	µg/L
4-Chlorotoluene	<5.00	-	-	-
1,2-Dibromo-3-chloropropane (DBCP)	<9.00	-	-	-
1,2-Dibromoethane (EDB)	23.0	4.36	18.4 - 28.1	15.0 - 31.1
Dibromomethane	51.4	2.73	41.9 - 63.2	33.4 - 69.4
1,2-Dichlorobenzene	<7.00	-	-	-
1,3-Dichlorobenzene	<7.00	-	-	-
1,4-Dichlorobenzene	<7.00	-	-	-
Dichlorodifluoromethane (Freon 12)	<5.00	-	-	-
1,1-Dichloroethane	42.9	0.816	33.7 - 52.8	27.2 - 58.9
1,2-Dichloroethane	<10.6	-	-	-
1,1-Dichloroethylene	23.8	3.92	16.2 - 31.9	14.1 - 35.0
cis-1,2-Dichloroethylene	<7.00	-	-	-
trans-1,2-Dichloroethylene	26.2	1.75	19.7 - 33.0	15.7 - 36.7
1,2-Dichloropropane	93.5	3.79	74.2 - 113	65.4 - 122
1,3-Dichloropropane	<5.00	-	-	-
2,2-Dichloropropane	<5.00	-	-	-
1,1-Dichloropropene	<5.00	-	-	-
cis-1,3-Dichloropropylene	60.8	4.07	45.3 - 72.4	39.5 - 82.1
trans-1,3-Dichloropropylene	42.2	3.07	32.4 - 51.9	27.4 - 57.0
Ethylbenzene	77.5	4.67	57.1 - 94.6	54.2 - 101
Hexachlorobutadiene	<4.30	-	-	-
Hexachloroethane	<3.30	-	-	-
2-Hexanone	36.9	2.33	23.4 - 49.4	13.6 - 58.2
Isopropylbenzene	<5.00	-	-	-
4-Isopropyltoluene	<5.00	-	-	-
Methylene chloride	40.8	0.812	29.7 - 51.4	24.5 - 57.1
4-Methyl-2-pentanone (MIBK)	47.0	2.61	31.5 - 61.6	20.4 - 71.8
Naphthalene	31.7	1.95	19.6 - 39.6	14.3 - 44.2
Nitrobenzene	<6.20	-	-	-
n-Propylbenzene	<5.00	-	-	-

Parameter	Certified Value[1]	Uncertainty[2]	QC Performance Acceptance Limits[3]	PT Performance Acceptance Limits[4]
	µg/L	%	µg/L	µg/L
Styrene	<13.0	-	-	-
1,1,1,2-Tetrachloroethane	47.6	5.94	37.3 - 58.5	30.9 - 64.3
1,1,2,2-Tetrachloroethane	42.7	6.23	32.9 - 54.7	27.8 - 57.6
Tetrachloroethylene	37.7	4.94	23.8 - 45.2	20.5 - 49.5
Toluene	34.5	3.32	26.6 - 41.1	24.2 - 44.8
1,2,3-Trichlorobenzene	<5.00	-	-	-
1,2,4-Trichlorobenzene	59.9	6.63	34.9 - 75.5	25.6 - 81.6
1,1,1-Trichloroethane	22.6	4.17	16.2 - 27.6	13.6 - 31.6
1,1,2-Trichloroethane	<10.5	-	-	-
Trichloroethylene	32.2	3.82	23.8 - 38.6	20.4 - 42.7
Trichlorofluoromethane	33.2	0.642	19.7 - 46.1	13.3 - 53.1
1,2,3-Trichloropropane (TCP)	29.8	4.05	18.4 - 40.2	11.5 - 46.4
1,2,4-Trimethylbenzene	20.1	6.23	15.4 - 25.1	13.1 - 27.1
1,3,5-Trimethylbenzene	83.9	7.38	65.3 - 104	54.5 - 113
Vinyl acetate	<5.00	-	-	-
Vinyl chloride	21.6	6.11	12.6 - 32.4	8.64 - 34.6
m-Xylene	47.5	0.814	-	-
m&p-Xylene	59.9	5.14	43.2 - 73.7	35.9 - 83.9
o-Xylene	23.6	5.60	17.0 - 29.0	14.2 - 33.0
p-Xylene	12.4	0.782	-	-
Xylenes, total	83.5	5.16	60.2 - 103	50.1 - 117

ANALYTICAL VERIFICATION

Parameter	Certified Value[1]	Proficiency Testing Study			NIST Traceability	
		Mean	Recovery[5]	n	SRM Number	Recovery
	µg/L	µg/L	%			%
Acetone	<3.90	-	-	-	-	-
Acetonitrile	<5.00	-	-	-	-	-
Acrolein	<5.00	-	-	-	-	-

Parameter	Certified Value[1]	Proficiency Testing Study		n	NIST Traceability	
		Mean	Recovery[5]	n	SRM Number	Recovery
	μg/L	μg/L	%			%
Acrylonitrile	<5.00	-	-	-	-	-
Benzene	68.2	67.9	99.5	14	1586-1	102
Bromobenzene	<5.00	-	-	-	-	-
Bromochloromethane	<5.00	-	-	-	-	-
Bromodichloromethane	<6.00	-	-	-	-	-
Bromoform	<6.00	-	-	-	-	-
Bromomethane	<8.00	-	-	-	-	-
2-Butanone (MEK)	<5.00	-	-	-	-	-
n-Butylbenzene	<5.00	-	-	-	-	-
sec-Butylbenzene	<5.00	-	-	-	-	-
tert-Butylbenzene	<5.00	-	-	-	-	-
tert-Butyl methyl ether (MTBE)	89.4	91.2	102	9	-	-
Carbon disulfide	<5.00	-	-	-	-	-
Carbon tetrachloride	16.9	15.7	92.8	12	1639	93.4
Chlorobenzene	94.1	93.0	98.9	14	1586-1	110
Chlorodibromomethane	<6.00	-	-	-	-	-
Chloroethane	<8.00	-	-	-	-	-
2-Chloroethylvinylether	<5.00	-	-	-	-	-
Chloroform	34.3	32.6	95.0	14	1639	105
Chloromethane	<8.00	-	-	-	-	-
2-Chlorotoluene	<5.00	-	-	-	-	-
4-Chlorotoluene	<5.00	-	-	-	-	-
1,2-Dibromo-3-chloropropane (DBCP)	<9.00	-	-	-	-	-
1,2-Dibromoethane (EDB)	23.0	23.5	102	10	-	-
Dibromomethane	51.4	51.0	99.3	9	-	-
1,2-Dichlorobenzene	<7.00	-	-	-	-	-
1,3-Dichlorobenzene	<7.00	-	-	-	-	-
1,4-Dichlorobenzene	<7.00	-	-	-	-	-
Dichlorodifluoromethane (Freon 12)	<5.00	-	-	-	-	-
1,1-Dichloroethane	42.9	37.8	88.1	14	-	-

Parameter	Certified Value[1]	Proficiency Testing Study		n	NIST Traceability	
		Mean	Recovery[5]	n	SRM Number	Recovery
	µg/L	µg/L	%			%
1,2-Dichloroethane	<10.6	-	-	-	-	-
1,1-Dichloroethylene	23.8	25.4	107	12	-	-
cis-1,2-Dichloroethylene	<7.00	-	-	-	-	-
trans-1,2-Dichloroethylene	26.2	27.0	103	12	-	-
1,2-Dichloropropane	93.5	96.2	103	14	3009	106
1,3-Dichloropropane	<5.00	-	-	-	-	-
2,2-Dichloropropane	<5.00	-	-	-	-	-
1,1-Dichloropropene	<5.00	-	-	-	-	-
cis-1,3-Dichloropropylene	60.8	60.0	98.7	14	-	-
trans-1,3-Dichloropropylene	42.2	42.2	100	14	-	-
Ethylbenzene	77.5	75.7	97.7	13	3002	98.7
Hexachlorobutadiene	<4.30	-	-	-	-	-
Hexachloroethane	<3.30	-	-	-	-	-
2-Hexanone	36.9	34.0	92.0	7	-	-
Isopropylbenzene	<5.00	-	-	-	-	-
4-Isopropyltoluene	<5.00	-	-	-	-	-
Methylene chloride	40.8	39.1	95.9	10	3008	105
4-Methyl-2-pentanone (MIBK)	47.0	44.4	94.5	7	-	-
Naphthalene	31.7	30.7	97.0	9	1647d	103
Nitrobenzene	<6.20	-	-	-	-	-
n-Propylbenzene	<5.00	-	-	-	-	-
Styrene	<13.0	-	-	-	-	-
1,1,1,2-Tetrachloroethane	47.6	46.1	96.8	9	-	-
1,1,2,2-Tetrachloroethane	42.7	42.5	99.4	12	-	-
Tetrachloroethylene	37.7	35.2	93.4	13	1639	105
Toluene	34.5	33.8	98.1	12	3001	103
1,2,3-Trichlorobenzene	<5.00	-	-	-	-	-
1,2,4-Trichlorobenzene	59.9	55.2	92.2	10	-	-
1,1,1-Trichloroethane	22.6	21.1	93.2	11	3011	95.6
1,1,2-Trichloroethane	<10.5	-	-	-	-	-
Trichloroethylene	32.2	31.6	98.1	13	1639	95.8

Parameter	Certified Value[1]	Proficiency Testing Study		n	NIST Traceability	
		Mean	Recovery[5]	n	SRM Number	Recovery
	µg/L	µg/L	%			%
Trichlorofluoromethane	33.2	37.3	112	10	-	-
1,2,3-Trichloropropane (TCP)	29.8	28.4	95.4	9	3014	97.1
1,2,4-Trimethylbenzene	20.1	21.9	109	9	-	-
1,3,5-Trimethylbenzene	83.9	80.8	96.3	9	-	-
Vinyl acetate	<5.00	-	-	-	-	-
Vinyl chloride	21.6	26.0	120	11	-	-
m-Xylene	47.5	-	-	-	3004	101
m&p-Xylene	59.9	62.7	105	9	-	-
o-Xylene	23.6	24.1	102	8	3003	99.2
p-Xylene	12.4	-	-	-	3005	102
Xylenes, total	83.5	84.4	101	10	-	-

1. The **Certified Values** are the actual "made-to" concentrations confirmed by ERA analytical verification. The certified values are monitored and purchasers will be notified of any significant changes resulting in recertification or withdrawal of this certified reference material during the period of validity of this certificate.

2. The **Uncertainty** is the total propagated uncertainty at the 95% confidence interval. The uncertainty is based on the preparation and internal analytical verification of the product by ERA, multiplied by a coverage factor. The uncertainty applies to the product as supplied and does not take into account any required or optional dilution and/or preparations the laboratory may perform while using this product.

3. The **QC Performance Acceptance Limits (QC PALs™)**are based on actual historical data collected in ERA's Proficiency Testing program. The QC PALs™ reflect any inherent biases in the methods used to establish the limits and closely approximate a 95% confidence interval of the performance that experienced laboratories should achieve using accepted environmental methods. Use the QC PALs™ to realistically evaluate your performance against your peers.

4. The **PT Performance Acceptance Limits (PT PALs™)** are calculated using the regression equations and fixed acceptance criteria specified in the NELAC proficiency testing requirements. Use the PT PALs™ when analyzing this QC standard alongside USEPA and NELAC compliant PT standards. Please note that many PT study acceptance limits are concentration dependent (some non-linearly) and, therefore, the acceptance limits of this QC standard and any PT standard may differ relative to their difference in concentrations.

5. The **PT Data/Traceability** data include the mean value, percent recovery and number of data points reported by the laboratories in our Proficiency Testing study compared to the Certified Values. In addition, where NIST Standard Reference Materials (SRMs) are available, each analyte has been analytically traced to the NIST SRM listed. This product is traceable to the lot numbers of its starting materials. All gravimetric and volumetric measurements related to its manufacture are traceable to NIST through an unbroken chain of comparisons.
Traceability Recovery (%) = [(% recovery certified standard)/(% recovery NIST SRM)]*100
The traceability data shown were compiled by analyzing the ERA standards or their associated stock solutions against the applicable NIST SRMs.

6. For additional information on this product such as intended use, instructions for use, level of homogeneity, and safety information, please refer to the provided Instruction Sheet

If you have any questions or need technical assistance, please call ERA technical assistance at 1-800-372-0122 or send an email to info@eraqc.com.

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NIST Special Publication 260-136

Standard Reference Materials

Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements

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FOREWORD

The National Institute of Standards and Technology (NIST), formerly the National Bureau of Standards, was established by the U.S. Congress in 1901 and charged with the responsibility for establishing a measurement foundation to facilitate both U.S. and international commerce. This charge was purposely stated in broad terms to provide NIST with the ability to establish and implement its programs in response to changes in national needs and priorities.

Increased requirements for quality systems documentation for trade and effective decision-making regarding the health and safety of the U.S. population have increased the need for demonstrating "traceability-to-NIST" and establishing a more formal means for documenting measurement comparability with standards laboratories of other nations and/or regions. Standard Reference Materials (SRM7s) are certified reference materials (CRMs) issued under NIST trademark that are well-characterized using state-of-the-art measurement methods for the determination of chemical composition and/or physical properties. SRMs are used to ensure the accuracy, traceability, and comparability of measurement results in many diverse fields of science, industry, and technology, both within the United States and throughout the world. The NIST Special Publication 260 Series is designed to provide details concerning the procedures and philosophy used at NIST to produce and certify SRMs and their appropriate use. A list of these publications can be accessed through the Internet at http://ts.nist.gov/srm.

This document provides definitions of the terms and a description of NIST's current practices for value-assigning SRMs and reference materials (RMs) used for calibrating and/or validating instrumentation and/or methods and procedures used for chemical measurements.

Willie E. May, Chief
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Technology Services

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Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurement

I. Introduction

Standard Reference Materials (SRMs) are certified reference materials (CRMs), issued under the National Institute of Standards and Technology (NIST) trademark that are well-characterized using state-of-the-art measurement methods and/or technologies for the determination of chemical composition and/or physical properties. Traditionally, SRMs have been the primary tools that NIST (formerly National Bureau of Standards) provides to the user community for achieving chemical measurement quality assurance and traceability to national standards.

This publication provides definitions of the terms and descriptions of NIST's current practices for value-assigning SRMs and reference materials (RMs) used for calibrating and/or validating instrumentation and/or methods and procedures used for chemical measurements. The terms and modes as described in this document are applicable for reference materials that support chemical measurements issued by NIST as of October 1, 1998.

Table 1 lists the seven modes used at NIST for value-assigning SRMs and RMs for chemical measurements and links the modes to three possible data quality descriptors: NIST Certified Values, NIST Reference Values, and NIST Information Values. A **NIST Certified Value** represents data for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST. A **NIST Reference Value** is a best estimate of the true value provided by NIST where all known or suspected sources of bias have not been fully investigated by NIST. A **NIST Information Value** is a value that will be of interest and use to the SRM/RM user, but insufficient information is available to assess the uncertainty associated with the value. Definitions of these modes are given in section IV.

Table 1. Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements

		NIST Certified Value	**NIST Reference Value**	**NIST Information Value**
1.	**Certification at NIST Using a Single Primary Method with Confirmation by Other Method(s)**	Y		
2.	**Certification at NIST Using Two Independent Critically-Evaluated Methods**	Y	Y	
3.	Certification/Value-Assignment **Using One Method at NIST** and Different Methods by Outside Collaborating Laboratories	Y	Y	
4.	Value-Assignment Based On Measurements by Two or More Laboratories Using Different Methods in Collaboration with NIST		Y	Y
5.	Value-Assignment Based on a Method-Specific Protocol		Y	Y
6.	Value-Assignment Based on NIST Measurements Using a Single Method or Measurements by an Outside Collaborating Laboratory Using a Single Method		Y	Y
7.	Value-Assignment Based on Selected Data from Interlaboratory Studies		Y	Y

The choice of mode(s) to be used in the value-assignment for any SRM for chemical measurements is based on our previous experiences and knowledge of the specific matrix, analyte(s) of interest, current measurement capabilities, the quality of the analytical methods results, and the intended use of the material.

The final designation of an assigned-value for an SRM as a NIST Certified Value, NIST Reference Value, or NIST Information Value is based on the specific value-assignment mode used and the assessed quality of the resulting data relative to the intended use of the material.

II. NIST Practices for Value-Assignment of SRMs and RMs for Chemical Measurements

Generally, NIST does not make or fabricate the materials from which SRMs are produced. Rather, U.S. industry, scientific groups, or companies on contract to NIST provide materials that meet NIST specifications.

Techniques and methods used at NIST for providing certified values for SRMs for chemical measurements are critically evaluated and have demonstrated accuracy in the matrix under investigation. Potential sources of error for such methods are evaluated and addressed [1,2]. Methods that are "ratio-based" (i.e., that require instrumental comparison versus calibrants of a known quantity of the measurand) use high-purity, well-characterized primary reference compounds or species as their basis for calibration (either directly or through gravimetrically prepared calibration solutions, e.g., NIST Elemental Solution SRMs).

The details of NIST methods and their testing are well documented (i.e., internal NIST Reports of Analysis) and often published in refereed technical journals. When results from outside laboratories are used in the value-assignment process, the NIST Chemical Science and Technology Laboratory (CSTL) is responsible for the selection of the laboratories and the technical evaluation of these reported data.

Appropriate control materials are concurrently analyzed in all value-assignment activities – both within NIST and by any outside collaborating laboratories. When available, appropriate SRMs or CRMs from other National Metrology Laboratories are used for this purpose.

III. Modes for Value-Assignment and/or Certification

The quality of assigned values for any CRM is based on the existence and application of sound measurement principles and practices. It is with this basic premise that we provide the following seven modes used at NIST to acquire analytical data for the value assignment of our SRMs and RMs for chemical measurements and link these modes to three data quality descriptors: NIST Certified Values, NIST Reference Values and NIST Information Values.

1. Certification at NIST Using a Single Primary Method with Confirmation by Other Method(s)

The Consultative Committee on the Quantity of Material (CCQM) [3] has described a primary method as:

> "A primary method of measurement is a method having the highest metrological properties, whose operation can be completely described and understood, for which a complete uncertainty statement can be written down in terms of SI units.
>
> > A primary direct method: measures the value of an unknown without reference to a standard of the same quantity.
> >
> > A primary ratio method: measures the value of a ratio of an unknown to a standard of the same quantity; its operation must be completely described by a measurement equation."

Certification at NIST using a single primary method is only possible when (with the exception of special cases noted below in 1.1, 1.2, and 1.3) [4]:

- All potentially significant sources of error have been evaluated explicitly for the application of the method and the matrix under investigation; a short written description is provided in the Report of Analysis for other sources of error that might reasonably be present and why they are not expected to be significant in this particular case.

- Confirmation of measurements by a primary NIST method can be accomplished by one or more of the following:
  - determination of certified constituents in other SRM(s) or CRM(s) of similar matrix and constituent concentration range;
  - a second NIST technique with appropriate controls; or
  - results of measurements from selected outside collaborating laboratories with appropriate experience.

The required level of agreement between the primary method and any confirmatory method(s) must be predetermined and specified in the experimental plan.

1.1. Certification of Gaseous Mixtures at NIST Using a Primary Method

Certification of gaseous mixture SRMs at NIST requires that:

- Primary standard suites be prepared gravimetrically from well-characterized starting materials and demonstrated to be internally consistent by a well-characterized analytical method.
- NIST primary standards be intercompared with primary standards from other National Metrology Laboratories or verified by a second NIST independent technique.
- Primary standards be documented to be stable for a minimum of two years.
- SRMs be value-assigned relative to the NIST primary standard suites.
- Uncertainty associated with the certified value includes contributions from the uncertainties associated with the primary standard suite, the analytical ratio method used to compare the SRM and primary standards, and the heterogeneity of the SRM lot.
    - The lot homogeneity is determined by NIST analysis of all samples.
- Absence of significant impurities in the SRMs be verified by NIST analyses.

1.2. Certification of NIST pH SRMs

Certification of NIST pH SRMs requires that:

- Homogeneity of the candidate material(s) for each pH SRM be evaluated by intercomparisons of randomly selected aliquots of candidate material(s) normalized to the preceding issuance of the corresponding pH SRM using a glass electrode.
- As an internal control measure, a candidate material be rejected if a significant difference is observed between the mean pH value of the current candidate material and the certified pH of the previous SRM issue (unless redetermination of the pH of the previous issue of the SRM using a Harned cell indicates a significant change from its certified pH value).
- Certification of each pH SRM be performed using cells without liquid junction (Harned cells) at each temperature of interest using at least three independently prepared buffer solutions of composite samples of the candidate SRM.
- Uncertainty associated with the certified value includes the measurement uncertainty (in potential of Harned cell), the uncertainty in standard potential of Ag, AgCl reference electrodes, the theoretical uncertainty in the conventional calculation of $-\log \gamma_{Cl}$, and the replication uncertainty for the overall pH value-assignment.

1.3. Certification of NIST Optical Filter SRMs

Specific requirements for the certification of NIST optical filter SRMs include:

1.3.1 Photometry

- Regular transmittance scale is maintained on the National Reference Spectrophotometer in the NIST Analytical Chemistry Division which is validated by the double-aperture method of light addition and benchmarked through international intercomparisons using optical filter artifact standards.

- Solid (neutral glass and metal-on-silica) SRMs are assigned certified values for transmittance and/or transmittance density at specified wavelengths by individual measurement of each artifact on the National Reference Spectrophotometer.

- Liquid or powder SRMs are assigned certified values for absorbance per unit pathlength or specific absorptivity at specified wavelengths by batch certification on the National Reference Spectrophotometer using a random sampling from the batch.

- The uncertainty for each assigned photometric value includes components to account for the precision and accuracy of the instrument; heterogeneity, temporal drift, and thermal characteristics of the artifact; and the geometry of sample positioning. Uncertainties are not individually evaluated but are based on pooled measurements with more than 30 degrees of freedom. Uncertainties are re-assessed annually for continuously produced solid standards or with each re-issue for batch-certified standards.

- A control filter is run with all data acquisitions, and the data are used to “control-chart" the measurement process and verify consistent performance.

1.3.2 Wavelength

- SRMs are assigned certified values for peak wavelength or wavenumber by comparison to atomic wavelengths (ultimately traceable to the standard meter) using a transfer spectrometer.

- Wavelength standards are batch certified using a random sampling from the lot. The uncertainty for each peak position in a wavelength standard includes components to account for the calibration accuracy of the transfer spectrometer, the precision in locating the standard peaks, and relevant temperature coefficients over the specified temperature range of valid certification.

2. Certification at NIST Using Two Independent Critically-Evaluated Methods

A second mode of certification for NIST SRMs involves the use of two or more critically evaluated independent methods [5,6]. Method independence is of critical importance, and while it is rare that two analytical methods have completely different sources of error and variability, they are chosen so that the most significant sources of error are different. For example, the following considerations are carefully evaluated:

- Methods are selected to minimize common steps in sample preparation and the final analytical measurement techniques.

- Methods rely on different physical, spectroscopic, or chemical phenomena that generate the analytical response.

- Methods/procedures selected are appropriate for the required precision and accuracy for measurement of the analyte(s) of interest in the matrix.

- The criteria for between-method agreement required for certification is pre-determined and documented in the experimental plan.

3. Certification/Value-Assignment Using One Method at NIST and Different Methods by Outside Collaborating Laboratories

In some cases, there does not exist a suitable second independent method at NIST. In these instances, we carefully select outside laboratories to collaborate on the certification process. Ideally this collaboration begins at the very start of the experimental design process. In this way, both NIST and outside laboratory analysts are able to coordinate the details of the measurement, data analysis, and reporting requirements for the SRM with careful attention to the following:

- The NIST method and the methods of the outside collaborating laboratories must have been critically evaluated and demonstrated to provide accurate results for the matrix under investigation.

- The method(s) used by outside collaborating laboratories should be different from the method used at NIST as required by the “Two Independent NIST Methods” mode (see Mode 2).

- Data reporting requirements for outside collaborating laboratories should be specified in the experimental plan, and reports should contain sufficient information to evaluate all significant sources of uncertainty.

4. Value-Assignment Based on Measurements by Two or More Laboratories Using Different Methods in Collaboration with NIST

This mode can be used to provide NIST Reference Values or NIST Information Values for an SRM, e.g., in instances in which there do not exist suitable methods at NIST. This mode requires that:

- The outside collaborating laboratories' methods have demonstrated accuracy in the matrix under investigation.

- Analyses provided by the outside collaborating laboratories involve at least two different methods (see Mode 2).

- Data reporting requirements for the outside collaborating laboratories be specified in the experimental plan and their report should contain sufficient information to evaluate all significant sources of uncertainty, unless a large number of labs/methods submit data, in which case the "Interlaboratory Study" criteria apply (see Mode 7).

5. Value-Assignment Based on a Method-Specific Protocol

In cases of method-defined parameters, the value of the parameters of interest result from the appropriate and validated use of a defined protocol. Appropriate implementation of this mode requires that:

- The protocol used be one that is recognized by the user community as the prescribed method for measurement of the analyte (or property) of interest in this matrix.

- Only data from experienced practitioners of the protocol be used.

- Measurements using the method-specific protocol be made by NIST, outside laboratories, or both.

- Method-specific value-assignment typically involve no fewer than three experienced practitioners of the method.

6. Value-Assignment Based on NIST Measurements Using a Single Method or Measurements by an Outside Collaborating Laboratory Using a Single Method

In some cases the intended use by the measurement community does not require a NIST Certified Value as an assigned value. This mode can be used to provide NIST Reference Values or NIST Information Values.

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- The NIST method used is typically one that would be used in the "Two Independent NIST Methods" mode (see Mode 2), i.e., the method may have been used in the past as one of several methods for SRM certification, but in this instance was the only method used.

- The method used by the outside laboratory must have been demonstrated to provide appropriate precision and accuracy in the matrix under investigation.

- Data reporting requirements for outside collaborating laboratories are specified in the experimental plan, and their reports should contain sufficient information to permit evaluation of significant sources of uncertainty.

7. Value-Assignment Based on Selected Data from Interlaboratory Studies

This mode allows NIST to take advantage of interlaboratory studies designed for purposes other than value-assignment of reference materials. In this mode:

- The particular study must be well documented and organized by a reputable organization.

- NIST Chemical Science and Technology Laboratory is responsible for evaluating the appropriateness of analytical procedures to identify a subset of results to be selected for use in value-assignment.

IV. Definition of Terms

NIST references a number of definitions in connection with the production, certification, and use of its SRMs and RMs. The uses of the terms "certified values," "reference values," etc., have multiple meanings based on the intent and practices of a particular reference material supplier. Certain definitions, adopted for NIST use, are derived from international guides and standards on reference materials and measurements while others have been developed by NIST to describe those activities unique to NIST operations and philosophy. To avoid any ambiguity, this publication provides definitions of the terms as they are currently used by NIST and a description of NIST's current practices for value-assigning SRMs and RMs that support chemical measurements. A listing of NIST-adopted and NIST-developed definitions follows.

Reference Material (RM): Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
[ISO VIM: 1993, 6.13 [7]*]*

Certified Reference Material (CRM): Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.
[ISO VIM: 1993, 6.14]

NIST Standard Reference Material® (SRM®): A CRM issued by NIST that also meets additional NIST-specified certification criteria. NIST SRMs are issued with Certificates of Analysis or Certificates that report the results of their characterizations and provide information regarding the appropriate use(s) of the material.

NIST Traceable Reference Material™ (NTRM™): A commercially-produced reference material with a well-defined traceability linkage to existing NIST standards for chemical measurements. This traceability linkage is established via criteria and protocols defined by NIST to meet the needs of the metrological community to be served.

NIST Certified Value: A value reported on an SRM Certificate/Certificate of Analysis for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST. Values are generally referred to as certified when Modes 1, 2, or 3 have been used for value-assignment and all the criteria for that mode are fulfilled. These three modes all require NIST measurements and oversight of the experimental design for the value-assignment process. The uncertainty associated with a certified value generally specifies a range within which the true value is expected to lie at a level of confidence of approximately 95 % if the sample is homogeneous. If significant sample heterogeneity is included, the uncertainty generally represents a prediction interval within which the true values of 95 % of all samples are expected to lie at a stated level of confidence.

Uncertainty of a Certified Value: An estimate attached to a certified value of a quantity which characterizes the range of values within which the "true value" is asserted to lie with a stated level of confidence. *[ISO Guide 30: 1992 3.4* [8]*]*

Uncertainty of a Measurement: Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand. *[ISO VIM: 1993 3.9]*

NIST Reference Value (formerly called Noncertified Value) for Chemical Composition and Related Properties: A NIST Reference Value is a best estimate of the true value provided on a NIST Certificate/Certificate of Analysis/Report of Investigation where all known or suspected sources of bias have not been fully investigated by NIST. Reference values are generally determined using the following modes:

- Mode 2 or 3 is used when there is lack of sufficient agreement among the multiple methods.

- Modes 4, 5, or 6 are used when the intended use of the value by the measurement community does not require that it be a certified value.

- Mode 7 can be used in special cases, e.g., when results are obtained from another national metrology laboratory with whom NIST has historical comparability data for the method(s) used for the specific matrix/analyte combination.

The uncertainty associated with a NIST Reference Value may not include all sources of uncertainty and may represent only a measure of the precision of the measurement method(s).

NIST Information Value: A NIST Information Value is considered to be a value that will be of interest and use to the SRM/RM user, but insufficient information is available to assess the uncertainty associated with the value. Typically, the information value has no reported uncertainty listed on the certificate and has been derived from one of the following value-assignment modes:

- Results from modes 4, 5, 6, or 7 in which the intended use of the value by the measurement community does not require that it be a certified or reference value. (E.g., information about the composition of the matrix such as the value of "total organic carbon" of a sediment material may be useful to the user in selecting an appropriate analytical method.)
- The results from modes 4, 5, 6, or 7 lack sufficient information to assess the uncertainty.
- Results are provided from outside NIST as supplemental information on the SRM matrix and are not measurements typically made at NIST but may be of interest to the user.

V. References

[1] Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, NIST Technical Note 1297, 1994 Edition.

[2] Guide to the Expression of Uncertainty of Measurement: First edition 1993; ISBN 92-67-10188-9; International Organization for Standardization (ISO), 1993.

[3] Minutes from the Fifth Meeting (February 1998) of the Consultative Committee on the Quantity of Material (CCQM) of the Bureau International des Poids et Mesures (BIPM), Sevres, France (1998).

[4] Definitive Measurement Methods, Moody, J.R., Epstein, M.S. Spectrochimica Acta, Vol. 46B, No. 12, (1991).

[5] The Independent Method Concept for Certifying Chemical Composition Reference Materials, Epstein, M.S., Spectrochimica Acta, Vol. 46B, No. 12, (1991).

[6] Combining Data From Independent Methods, Schiller, S.B. and Eberhardt, K.B., Spectrochimica Acta, Vol. 46B, No. 12, (1991).

[7] International Vocabulary of Basic and General Terms in Metrology (VIM), 2nd Edition; BIPM/IEC/IFCC/ISO/IUPAC/IUPAP/OIML, International Organization for Standardization (ISO), 1993.

[8] Terms and Definitions Used in Connection with Reference Materials, ISO Guide 30, International Organization for Standardization (ISO), 1992.

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2709a

San Joaquin Soil

Baseline Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. One unit of SRM 2709a consists of 50 g of dried, powdered, agricultural soil.

Certified Values: The certified concentrations for 19 elements, expressed as mass fractions [1] on a dry-mass basis, are provided in Table 1. Certified values are based on results obtained from critically evaluated independent analytical techniques. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [2].

Reference Values: The reference values for 15 constituents, expressed as mass fractions on a dry-mass basis, are provided in Table 2. The reference values are based on results obtained from a single NIST analytical method. Reference values are non-certified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty [2].

Information Values: The values for 10 elements are provided in Table 3 for information purposes only. These are non-certified values with no uncertainty assessed. The information values included in this certificate are based on results obtained from one NIST method.

Expiration of Certification: The certification of SRM 2709a is valid, within the measurement uncertainties specified, until **1 November 2018**, provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

E.A. Mackey and R.R. Greenberg of the NIST Analytical Chemistry Division were responsible for coordination of the technical measurements leading to certification.

Statistical analyses were performed by J.H. Yen of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 7 April 2009

Robert L. Watters, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR USE

Sampling: The SRM should be thoroughly mixed by repeatedly inverting and rotating the bottle horizontally before removing a test portion for analysis. A minimum mass of 250 mg (dry mass - see *Instructions for Drying*) should be used for analytical determinations to be related to the mass fraction values in this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to effect complete dissolution. If volatile elements (i.e., arsenic, mercury, selenium) are to be determined, precautions should be taken in the dissolution of SRM 2709a to avoid volatilization losses.

Drying: To relate measurements to the certified, reference, and information values that are expressed on a dry-mass basis, users should determine a drying correction at the time of each analysis. The recommended drying procedure is oven drying for 2 h at 110 °C. Note that analytical determination of volatile elements (i.e., arsenic, mercury, selenium) should be determined on samples as received; separate samples should be dried as previously described to obtain a correction factor for moisture. Correction for moisture is to be made to the data for volatile elements before comparing them to the certified values. This procedure ensures that these elements are not lost during drying. The mass loss on drying for this material as bottled was approximately 3%, but this value may change once the bottle is opened and the soil is exposed to air.

SOURCE, PREPARATION, AND ANALYSIS

Source and Preparation of Material[1]: The U.S. Geological Survey (USGS), under contract to NIST, collected and processed the soil for SRM 2709a with assistance from the U.S. Bureau of Reclamation's Sacramento CA office. The agricultural soil used to produce SRM 2709a was collected from a fallow field, in the central California San Joaquin Valley. Three separate collection sites were used to obtain the necessary amount of material. Each collection site covered an area of approximately 4 m^2. Prior to sample collection the area was scraped clean of surface vegetation. Collected material was transferred to 20 plastic-lined five-gallon plastic buckets and shipped to the USGS laboratory for processing. At USGS, the SRM 2709a soil was dried at room temperature, disaggregated, and sieved to remove coarse material (≥2 mm). The resulting soil was ball-milled in 50 kg portions, and then the entire batch of soil was transferred to a cross-flow V-blender for mixing. The blended soil was radiation sterilized prior to bottling. In the final preparation step the blended material was split into containers using a custom-designed spinning riffler, which was used to divide the material into smaller batches, and then used to apportion approximately 50 g into each pre-cleaned bottle.

Every 100th bottle was set aside for chemical analyses designed to assess material homogeneity using X-ray fluorescence spectrometry (XRF), inductively coupled plasma optical emission spectrometry (ICP-OES), and inductively coupled plasma mass spectrometry (ICP-MS) at the USGS. Homogeneity assessments were performed at NIST as well, and results indicated that additional processing was needed to achieve optimum homogeneity. The material from all bottles was combined, and then ground in batches between stainless steel plates for a time sufficient to produce a powder of which ≥95%, by mass, passed through a 200 mesh (74 µm) sieve. The resulting powder was blended, and 50 g portions were dispensed into bottles using the spinning riffler. Results from additional analyses indicated material homogeneity was acceptable (see below).

Analysis: The homogeneity was assessed for selected elements in the bottled material using X-ray fluorescence spectrometry and instrumental neutron activation analysis. In a few cases, statistically significant differences were observed, and the variance due to material inhomogeneity is included in the expanded uncertainties of the certified values. The estimated relative standard deviation for material inhomogeneity for most elements is ≤1 %, for calcium it is approximately 2 %, and for chromium it is approximately 3 %. Significant material heterogeneity was observed for mercury, for which a reference value with a prediction interval is provided; see Table 2.

Analyses of this material were performed at NIST (Gaithersburg, MD) and at the USGS (Denver, CO). Results from NIST were used to provide the certified, reference, and information values shown in Tables 1, 2, and 3 respectively. Results from the USGS were used to confirm those values. The analytical techniques used for each element are listed in Table 4; the analysts are listed in Tables 5 and 6.

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[1] Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 1. Certified Values[(a)] (Dry-Mass Basis) for Selected Elements in SRM 2709a

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Aluminum	7.37 ± 0.16	Antimony	1.55 ± 0.06
Calcium	1.91 ± 0.09	Barium	979 ± 28
Iron	3.36 ± 0.07	Cadmium	0.371 ± 0.002
Magnesium	1.46 ± 0.02	Chromium	130 ± 9
Phosphorus	0.0688 ± 0.0013	Cobalt	12.8 ± 0.2
Potassium	2.11 ± 0.06	Lead	17.3 ± 0.1
Silicon	30.3 ± 0.4	Manganese	529 ± 18
Sodium	1.22 ± 0.03	Strontium	239 ± 6
Titanium	0.336 ± 0.007	Vanadium	110 ± 11
		Zirconium	195 ± 46

(a) Certified values for all elements except cadmium and lead are the equally weighted means of results from two or three analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor determined by the Student's t distribution, calculated by combining a between-method variance with a pooled within-method variance [3] following the ISO Guide [4]. A component for material heterogeneity is incorporated into the uncertainties of aluminum, calcium, chromium, manganese, and sodium. The certified values for cadmium and lead are each results from a single NIST method (isotope dilution (ID)-ICP-MS) for which a complete evaluation of all sources of uncertainty has been performed. The uncertainty for the certified value for cadmium and lead represents an expanded uncertainty with a coverage factor of 2, with uncertainty components combined following the ISO Guide [4].

Table 2. Reference Values[(a)] (Dry-Mass Basis) for Selected Elements in SRM 2709a

Element	Mass Fraction (mg/kg)
Arsenic	10.5 ± 0.3
Cerium	42 ± 1
Cesium	5.0 ± 0.1
Copper	33.9 ± 0.5
Europium	0.83 ± 0.02
Gadolinium	3.0 ± 0.1
Lanthanum	21.7 ± 0.4
Mercury[(b)]	0.9 ± 0.2
Nickel	85 ± 2
Rubidium	99 ± 3
Scandium	11.1 ± 0.1
Thallium	0.58 ± 0.01
Thorium	10.9 ± 0.2
Uranium	3.15 ± 0.05
Zinc	103 ± 4

(a) Reference values for all elements are based on results from one analytical method at NIST. Uncertainty values represent the expanded uncertainties which include the combined Type A and Type B with a coverage factor of 2, following the ISO Guide [4].

(b) The reference value for mercury represents the average value from analysis of portions from six bottles using ID-cold vapor (CV)-ICP-MS. Results indicate significant material heterogeneity; values ranged from 0.8 mg/kg to 1.1 mg/kg. The uncertainty for this reference value is in the form of a prediction interval.

Table 3. Information Values[(a)] (Dry Mass Basis) for Selected Elements in SRM 2709a

Element	Mass Fraction (mg/kg)
Boron	74
Dysprosium	3
Hafnium	4
Lutetium	0.3
Neodymium	17
Samarium	4
Selenium	1.5
Tantalum	0.7
Terbium	0.5
Ytterbium	2

(a) Information values are based on results from one analytical method at NIST.

Table 4. Methods Used for the Analysis of SRM 2709a

Element	Certification Methods
Al	INAA; XRF
As	INAA
B	PGAA
Ba	ICP-OES; INAA: XRF
Ca	INAA; XRF
Cd	ID-ICP-MS; PGAA
Ce	INAA
Co	INAA; ICP-OES
Cr	INAA; XRF
Cs	INAA
Cu	ICP-MS
Dy	INAA
Eu	INAA
Fe	INAA; PGAA; XRF
Gd	PGAA
Hf	INAA
Hg	CV-ID-ICP-MS
K	INAA; PGAA; XRF
La	INAA
Lu	INAA
Mg	INAA; XRF
Mn	INAA; PGAA; XRF
Na	INAA; XRF
Nd	INAA
Ni	ICP-MS
P	ICP-OES; XRF
Pb	ID-ICP-MS
Rb	INAA
Sb	INAA; ICP-MS
Sc	INAA
Se	CCT-ICP-MS
Si	PGAA; XRF
Sm	INAA
Sr	INAA; ICP-OES; XRF
Ta	INAA
Tb	INAA
Th	INAA
Ti	INAA; PGAA; XRF
Tl	ICP-MS
U	ICP-MS
V	INAA; XRF
Yb	INAA
Zn	INAA
Zr	INAA; XRF

NIST Methods of Analysis

CCT-ICP-MS	Collision Cell Inductively Coupled Plasma Mass Spectrometry
CV ID-ICP-MS	Cold Vapor Isotope Dilution Inductively Coupled Plasma Mass Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
ID-ICP-MS	Isotope Dilution Inductively Coupled Plasma Mass Spectrometry
INAA	Instrumental Neutron Activation Analysis
PGAA	Prompt Gamma-Ray Activation Analysis
XRF	X-ray Fluorescence Spectrometry

USGS Methods of Analysis(a)

WD-XRF	Wavelength Dispersive X-ray Fluorescence Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry

(a) USGS Methods of Analysis were used to confirm results from certification methods.

Table 5. Participating NIST Analysts:

S.J. Christopher
R.M. Lindstrom
S.E. Long
E.A. Mackey
A.F. Marlow
K.E. Murphy
R.L. Paul
S.A. Rabb
J.R. Sieber
R.O. Spatz
R.S. Popelka-Filcoff
B.E. Tomlin
L.J. Wood
L.L. Yu
R. Zeisler

Table 6. Participating USGS Laboratory and Analysts

Laboratory	Analysts
U.S. Geological Survey Branch of Geochemistry Denver, CO, USA	M.G. Adams Z.A. Brown P.L. Lamothe J.E. Taggart S.A. Wilson

REFERENCES

[1] Thompson, A; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*, NIST Special Publication 811 (2008); available at http://physics.nist.gov/Pubs/

[2] May, W.E.; Gills, T.E.; Par*ris, R.; Beck, II, C.M.; Fassett, J.D.; Gettings, R.J.; Gre*enberg, R.R.; Guenther, F.R.; Kramer, G.; MacDonald, B.S.; Wise, S.A.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*, NIST Special *Publication 260-136* (1999); available at http://www.cstl.nist.gov/nist839/NIST_special_publications.htm).

[3] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; J. Res. NIST 105, pp. 571-579 (2000).

[4] ISO; *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

Addendum to Certificate

Standard Reference Material® 2709a

San Joaquin Soil

Baseline Trace Element Concentrations

Leachable Concentrations Determined Using USEPA Methods 200.7 and 3050B

The mass fraction values contained in the NIST Certificate of Analysis for SRM 2709a represent the total element content of the material. The measurement results used to provide the certified, reference, or information values are obtained from methods that require complete sample decomposition, or from nondestructive analytical methods such as instrumental neutron activation analysis or prompt gamma-ray activation analysis. Where complete sample decomposition is required, it can be accomplished by digestion with mixed acids or by fusion. For mixed-acid decomposition, hydrofluoric acid must be included in the acid mixture used to totally decompose siliceous materials such as soils and sediments.

In its monitoring programs, the U.S. Environmental Protection Agency (USEPA) has established a number of leach methods for the preparation of soil samples for the determination of extractable elements. Eight laboratories participated, seven of which used USEPA Method 200.7; the remaining laboratory used USEPA SW-846 Method 3050B for preparation of soil samples. All elements were determined in leachates by inductively coupled plasma optical emission spectrometry. Six of the eight laboratories provided individual results from duplicate portions, and these results were averaged together to provide one result for each element from each participating laboratory. Results rejected as outliers by the USEPA Contract Laboratory Program (CLP) officials were not included. Results are summarized in Table A1. The ranges of mass fraction values, median values (to two significant figures), and the number of results included for each are given for 23 elements. The percent recovery values based on the ratios of the median values to the total element content (from the certified, reference, or information values in the Certificate of Analysis) are listed in the last column of Table A1. **Note that the certified values provided as total mass fractions in the Certificate of Analysis are the best estimate of the true mass fraction values for this material.**

This USEPA CLP Study was coordinated by Clifton Jones, Quality Assurance and Technical Support Program, Shaw Environmental & Infrastructure Group, Las Vegas, NV, under the direction of John Nebelsick, USEPA, Analytical Services Branch. The participating laboratories are listed in Table A2.

Table A1. Results from Laboratories Participating in the EPA Contract Laboratory Program Study.

Element	n	Range (mg/kg)			Median (mg/kg)	Recovery (%)
Aluminum	7	13000	-	17000	16000	22
Antimony	2	1.2	-	1.5	1.4	88
Arsenic	8	6.4	-	10	7.8	74
Barium	8	350	-	400	380	39
Beryllium	7	0.50	-	0.72	0.61	--
Cadmium	5	0.33	-	0.66	0.40	110
Calcium	8	12000	-	14000	12000	65
Chromium	8	46	-	67	53	41
Cobalt	8	8.2	-	13	10	81
Copper	7	24	-	28	27	81
Iron	8	22000	-	26000	24000	70
Lead	7	8.1	-	11	9.2	53
Magnesium	7	9700	-	11000	10000	71
Manganese	8	380	-	450	420.0	79
Mercury	8	0.79	-	0.92	0.87	97
Nickel	8	59	-	71	66	77
Potassium	8	2600	-	4000	2900	14
Selenium	5	0.69	-	1.9	0.95	63
Silver	4	0.14	-	4.1	0.64	--
Sodium	7	460	-	610	500	4
Thallium	2	0.74	-	1.6	1.2	200
Vanadium	8	43	-	71	48	44
Zinc	8	69	-	87	79	77

Table A2. List of CLP and non-CLP Participating Laboratories

A4 Scientific, Inc.
Bonner Analytical Testing Co.
Chem Tech Consulting Group
Datachem Laboratories, Inc.
Liberty Analytical Corporation
MSE Laboratory Services
Shealy Environmental
SVL Analytical Inc.

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1944

New York/New Jersey Waterway Sediment

Standard Reference Material (SRM) 1944 is a mixture of marine sediment collected near urban areas in New York and New Jersey. SRM 1944 is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and trace elements in marine sediment and similar matrices. Reference values are also provided for selected polybrominated diphenyl ether (PBDE) congeners, selected dibenzo-*p*-dioxin and dibenzofuran congeners, total organic carbon, total extractable material, and particle size characteristics. Information values are provided for selected polychlorinated naphthalenes (PCNs) and hexabromocyclododecanes (HBCDs). All of the constituents for which certified, reference, and information values are provided in SRM 1944 were naturally present in the sediment before processing. A unit of SRM 1944 consists of a bottle containing 50 g of radiation-sterilized, freeze-dried sediment.

Certified Mass Fraction Values: Certified values for mass fractions of PAHs, PCB congeners, chlorinated pesticides, and trace elements are provided in Tables 1 through 4. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST using two or more chemically independent analytical techniques. The certified values for the trace elements are based on NIST measurements by one technique and additional results from several collaborating laboratories.

Reference Mass Fraction Values: Reference values are provided for mass fractions of additional PAHs (some in combination) in Tables 5 and 6, additional PCB congeners and chlorinated pesticides in Table 7, PBDE congeners in Table 8, and additional inorganic constituents in Tables 9 and 10. Reference values are provided in Table 11 for the 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and total tetra-, penta-, hexa-, and hepta-congeners of polychlorinated dibenzo-*p*-dioxin and dibenzofuran. Reference values for particle size characteristics are provided in Table 12 and 13. Reference values for total organic carbon and percent extractable mass are provided in Table 14. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1].

Information Mass Fraction Values: Information values are provided in Table 15 for mass fractions of additional trace elements, in Table 16 for PCN congeners (some in combination), and in Table 17 for HBCD isomers. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [1].

Expiration of Certification: The certification of **SRM 1944** is valid, within the measurement uncertainties specified, until **31 March 2017**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Stephen A. Wise, Chief
Analytical Chemistry Division

Robert L. Watters, Jr., Chief
Measurement Services Division

Gaithersburg, MD 20899
Certificate Issue Date: 27 September 2011
Certificate Revision History on Page 20

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification was performed by M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh, M.G. Vangel, and M.S. Levenson of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

The sediment was collected with the assistance of the New York District of the U.S. Army Corp of Engineers (ACENYD), who provided the expertise in the site selection, the ship, sampling equipment, and personnel. L. Rosman of ACENYD and R. Parris (NIST) coordinated the collection of this sediment. Collection and preparation of SRM 1944 were performed by R. Parris, M. Cronise, and C. Fales (NIST); L. Rosman and P. Higgins (ACENYD), and the crew of the *Gelberman* from the ACE Caven Point facility in Caven Point, NJ.

Analytical measurements for the certification of SRM 1944 were performed at NIST by E.S. Beary, D.A. Becker, R.R. Greenberg, J.M. Keller, J.R. Kucklick, M. Lopez de Alda, K.E. Murphy, R. Olfaz, B.J. Porter, D.L. Poster, L.C. Sander, P. Schubert, M.M. Schantz, S.S. Vander Pol, and L. Walton of the Analytical Chemistry Division. Measurements for percent total organic carbon measurements were provided by three commercial laboratories and T.L. Wade of the Geochemical and Environmental Research Group, Texas A&M University (College Station, TX, USA). The particle-size distribution data were provided by Honeywell, Inc. (Clearwater, FL, USA). Additional results for PBDE congeners were used from ten laboratories (see Appendix A) that participated in an interlaboratory study specifically for PBDEs in Marine Sediment coordinated by H.M. Stapleton of the NIST Analytical Chemistry Division. M. LaGuardia of Virginia Institute of Marine Science (Gloucester Point, VA, USA) provided one set of measurements for the HBCDs.

Values for the polychlorinated dibenzo-*p*-dioxins and dibenzofurans were the results of an interlaboratory comparison study among fourteen laboratories (see Appendix B) coordinated by S.A. Wise of the NIST Analytical Chemistry Division and R. Turle and C. Chiu of Environment Canada Environmental Technology Centre, Analysis and Air Quality Division (Ottawa, ON, Canada). Analytical measurements for selected trace elements were provided by the International Atomic Energy Agency (IAEA, Seibersdorf, Austria) by M. Makarewicz and R. Zeisler. Results were also used from seven laboratories (see Appendix C) that participated in an intercomparison exercise coordinated by S. Willie of the Institute for National Measurement Standards, National Research Council Canada (NRCC; Ottawa, ON, Canada).

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This material is naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

Storage: SRM 1944 must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Use: Prior to removal of test portions for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 1944 are reported on a dry-mass basis. The SRM, as received, contains a mass fraction of approximately 1.3 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis or, if the constituents of interest are volatile, a separate test portion of the sediment should be removed from the bottle at the time of analysis and dried to determine the mass fraction on a dry-mass basis.

PREPARATION AND ANALYSIS[1]

Sample Collection and Preparation: The sediment used to prepare this SRM was collected from six sites in the vicinity of New York Bay and Newark Bay in October 1994. Site selection was based on contaminant levels measured in previous samples from these sites and was intended to provide relatively high concentrations for a variety of chemical classes of contaminants. The sediment was collected using an epoxy-coated modified Van Veen-type grab sampler designed to sample the sediment to a depth of 10 cm. A total of approximately 2100 kg of wet sediment was collected from the six sites. The sediment was freeze-dried, sieved (nominally 250 µm to 61 µm), homogenized in a cone blender, radiation sterilized at an estimated minimum dose of 32 kilograys (^{60}Co), and then packaged in screw-capped amber glass bottles.

Conversion to Dry-Mass Basis: The results for the constituents in SRM 1944 are reported on a dry-mass basis; however, the material as received contains residual moisture. The amount of moisture in SRM 1944 was determined by measuring the mass loss after freeze drying test portions of 1.6 g to 2.5 g for five days at 1 Pa with a –10 °C shelf temperature and a –50 °C condenser temperature. The mass fraction of moisture in SRM 1944 at the time of the certification analyses was 1.25 % ± 0.03 % (95 % confidence level).

Polycyclic Aromatic Hydrocarbons: The general approach used for the value assignment of the PAHs in SRM 1944 consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques [2]. Techniques and solvents involved were Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, clean up of the extracts using solid-phase extraction (SPE), or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) analysis of the total PAH fraction, (2) reversed-phase LC-FL analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC), (3) gas chromatography/mass spectrometry (GC/MS) analysis of the PAH fraction on four stationary phases of different selectivity, i.e., a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase, a 50 % phenyl-substituted methylpolysiloxane phase, a proprietary non-polar polysiloxane phase, and a smectic liquid crystalline stationary phase.

Seven sets of GC/MS results, designated as GC/MS (I), GC/MS (II), GC/MS (III), GC/MS (IV), GC/MS (V), GC/MS (VI), and GC/MS (Sm), were obtained using four columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate test portions of 1 g from eight bottles of SRM 1944 were Soxhlet extracted for 24 h with DCM. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 2 % DCM in hexane. (All extraction and LC solvent compositions are expressed as volume fractions unless otherwise noted.) The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using 1 g to 2 g test portions from three bottles of SRM 1944 and 2 g to 3 g test portions from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment). These test portions were Soxhlet extracted with DCM and processed through the silica SPE as described above; however, the extract was further fractionated using normal-phase LC on a semi-preparative aminopropylsilane column to isolate the PAH fraction. The PAH fraction was then analyzed using the same column as described above for GC/MS (I); however, the test portions were extracted, processed, and analyzed as part of three different sample sets at different times using different calibrations for each set. For the GC/MS (III), 1 g to 2 g test portions from six bottles of SRM 1944 were Soxhlet extracted for 18 h with 250 mL of a mixture of 50 % hexane/50 % acetone. The extracts were then processed and analyzed as described for GC/MS (II). For GC/MS (IV) analyses, 1 g to 2 g test portions from six bottles of SRM 1944 were extracted using PFE with a mixture of 50 % hexane/50 % acetone, and the extracts were processed as described above for GC/MS (II). The GC/MS (V) results were obtained by analyzing three of the same PAH fractions that were analyzed in GC/MS (III) and three of the PAH fractions that were analyzed in GC/MS (IV) using a 50 % (mole fraction) phenyl-substituted methylpolysiloxane stationary phase (0.25 mm i.d. × 60 m, 0.25 µm film thickness) (DB-17MS, J&W Scientific, Folsom, CA). For GC/MS (VI) analyses, three test portions of 0.7 g from one bottle of SRM 1944 were Soxhlet extracted for 24 h with DCM. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 20 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a proprietary non-polar polysiloxane phase (0.25 µm film thickness) (DB-XLB, J&W Scientific). For GC/MS (Sm) 1 g to 2 g test portions from six bottles of SRM 1944 were Soxhlet extracted for 24 h with 250 mL of DCM. The extracts were processed as described above for

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(1) Certain commercial equipment, instruments, or materials are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

GC/MS (I) using an aminopropylsilane SPE cartridge followed by GC/MS analysis using 0.2 mm i.d. × 25 m (0.15 µm film thickness) smectic liquid crystalline phase (SB-Smectic, Dionex, Lee Scientific Division, Salt Lake City, UT).

Two sets of LC-FL results, designated as LC-FL (Total) and LC-FL (Fraction), were used in the certification process. Test portions of approximately 1 g from six bottles of SRM 1944 were Soxhlet extracted for 20 h using 200 mL of 50 % hexane/50 % acetone. The extracts were concentrated and then processed through two aminopropylsilane SPE cartridges connected in series to obtain the total PAH fraction. A second 1 g test portion from the six bottles was Soxhlet extracted and processed as described above; the PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (µBondapak NH_2, 9 mm i.d. × 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions. The total PAH fraction and the isomeric PAH fractions were analyzed using a 5-µm particle-size polymeric octadecylsilane (C_{18}) column (4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength-programmed fluorescence detection. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1944 was assessed by analyzing duplicate test portions of 1 g from eight bottles selected by stratified random sampling. Test portions were extracted, processed, and analyzed as described above for GC/MS (I). No statistically significant differences among bottles were observed for the PAHs at the 1 g test portion size.

PAH Isomers of Molecular Mass 300 and 302: For the determination of the molecular mass 300 and 302 PAH isomers, three test portions of approximately 5 g each were extracted using PFE with DCM. The extracts were then concentrated with a solvent change to hexane and passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substitued methylpolysiloxane phase (0.25 µm film thickness; DB-17MS, J&W Scientific, Folsom, CA). Perdeuterated dibenzo[*a,i*]pyrene was added to the sediment prior to extraction for use as an internal standard.

PCBs and Chlorinated Pesticides: The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1944 consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques [2]. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, clean up/isolation using SPE or LC, followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity.

Eight sets of results were obtained designated as GC-ECD (I) A and B, GC-ECD (II) A and B, GC/MS (I), GC/MS (II), GC/MS (III), and QA Exercise. For the GC-ECD (I) analyses, 1 g test portions from four bottles of SRM 1944 were Soxhlet extracted with DCM for 18 h. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The concentrated eluant was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides and, (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.32 mm × 100 m fused silica capillary column with a 50 % (mole fraction) octadecyl (C18) methylpolysiloxane phase (0.1 µm film thickness) (CPSil 5 C18 CB, Chrompack International, Middelburg, The Netherlands). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the C18 phase are designated as GC-ECD (IB). A second set of samples was also analyzed by GC-ECD (i.e., GC-ECD IIA and IIB). Test portions of 1 g to 2 g from three bottles of SRM 1944 and 2 g to 3 g test portions from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment) were extracted, processed, and analyzed as described above for GC-ECD (I); however, the test portions were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set.

Three sets of results were obtained by GC/MS. For GC/MS (I), 1 g to 2 g test portions from six bottles were Soxhlet extracted with a mixture of 50 % hexane/50 % acetone. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The extract was then analyzed by GC/MS using a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness). The GC/MS (II) results were obtained in the same manner as the GC/MS (I) analyses except that the six test portions were extracted using PFE. The GC/MS (III) analyses were performed on the same extract fractions analyzed in GC-ECD (II) using the 5 % phenyl-substituted methylpolysiloxane phase describe above for GC/MS (I). For both the GC-ECD and GC/MS analyses, two PCB congeners that are not significantly present in the sediment extract (PCB 103 and PCB 198 [3]), and 4,4'-DDT-$\boldsymbol{d}_8$ were added to the sediment prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1944 was used in an interlaboratory comparison exercise in 1995 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [4]. Results from nineteen laboratories that participated in this exercise were used as the eighth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1944. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure PCB congeners and chlorinated pesticides.

Polybrominated Diphenyl Ethers: Value assignment of the concentrations of eight PBDE congeners was based on the means of results from two interlaboratory studies [5,6] and two sets of data from NIST. The laboratories participating in the interlaboratory exercises (see Appendix A) employed the analytical procedures routinely used in their laboratories to measure PBDEs. For the two methods used at NIST, six test portions (between 1 g and 2 g) were extracted using PFE at 100 °C with DCM. The extracts were cleaned up using an alumina column (5 % deactivated) SPE column. Size exclusion chromatography (SEC) on a divinylbenzene-polystyrene column (10 µm particle size, 10 nm (100 angstrom) pore size, 7.5 mm i.d. × 300 mm, PL-Gel, Polymer Labs, Inc.) was then used to remove the sulfur. The PBDEs, as well as PCBs and pesticides, were quantified using GC/MS in the electron impact mode on a 0.18 mm i.d. × 30 m fused silica capillary column with a 5 % (mole fraction) phenyl methylpolysiloxane phase (0.18 µm film thickness; DB-5MS, Agilent Technologies). The PBDEs were also quantified using GC/MS in the negative chemical ionization mode on a 0.18 mm i.d. × 10 m fused silica capillary column with a 5 % (mole fraction) phenyl methylpolysiloxane phase (0.18 µm film thickness; DB-5MS, Agilent Technologies). Selected Carbon-13 labeled PBDE and PCB congeners were added to the sediment prior to extraction for use as internal standards for quantification purposes.

Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans: Value assignment of the concentrations of the polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and the total tetra- through hepta- substituted polychlorinated dibenzo-*p*-dioxins and dibenzofurans was accomplished by combining results from the analysis of SRM 1944 by fourteen laboratories that participated in an interlaboratory comparison study (see Appendix B). Each laboratory analyzed three test portions (typically 1 g) of SRM 1944 using their routine analytical procedures and high resolution gas chromatography with high resolution mass spectrometry detection (GC-HRMS). The analytical procedures used by all of the laboratories included spiking with ^{13}C-labeled surrogates (internal standards); Soxhlet extraction with toluene; sample extract cleanup with acid/base silica, alumina, and carbon columns; and finally analysis of the cleaned up extract with GC-HRMS. Most of the laboratories used a 5 % phenyl-substituted methylpolysiloxane phase capillary column (DB-5), and about half of the laboratories confirmed 2,3,7,8-tetrachlorodibenzofuran using a 50 % cyanopropylphenyl-substituted methylpolysiloxane (DB-225, J&W Scientific, Folsom, CA) capillary column.

Analytical Approach for Inorganic Constituents: Value assignment for the concentrations of selected trace elements was accomplished by combining results of the analyses of SRM 1944 from NIST, NRCC, IAEA, and seven laboratories that participated in an interlaboratory comparison exercise coordinated by NRCC [7] (see Appendix C). The analytical methods used for the determination of each element are summarized in Table 18. For the certified concentration values listed in Table 4, results were combined from: (1) analyses at NIST using isotope dilution inductively coupled plasma mass spectrometry (ID-ICPMS) or instrumental neutron activation analysis (INAA), (2) analyses at NRCC using ID-ICPMS, graphite furnace atomic absorption spectrometry (GFAAS), and/or inductively coupled plasma optical emission spectroscopy (ICPOES), (3) analyses at IAEA using INAA, and (4) the mean of the results from seven laboratories that participated in the NRCC interlaboratory comparison exercise. The reference mass fraction values in Table 9 were determined by combining results from (1) analyses performed at NIST using INAA; (2) analyses at NRCC using ID-ICPMS, GFAAS, ICPOES, and/or cold vapor atomic absorption spectroscopy (CVAAS); (3) analyses at IAEA using INAA; and (4) the mean of the results from five to seven laboratories that participated in the NRCC interlaboratory comparison exercise. The information concentration values in Table 15 were determined by INAA at NIST and IAEA.

NIST Analyses using ID-ICPMS: Lead, cadmium, and nickel were determined by ID-ICPMS [8]. Test portions (0.4 g to 0.5 g) from six bottles of the SRM were spiked with ^{206}Pb, ^{111}Cd, and ^{62}Ni and wet ashed using a combination of nitric, hydrochloric, hydrofluoric, and perchloric acids. Lead and cadmium were determined in the same test portions; nickel was determined in a second sample set. A small amount of crystalline material remained after the acid dissolution. Lithium metaborate fusion was performed on this residue to confirm that the residue contained insignificant amounts of the analytes. Cadmium and nickel were separated from the matrix material to eliminate the possibility of spectral interferences, and concentrations were determined from the measurement of the $^{112}Cd/^{111}Cd$ and $^{62}Ni/^{60}Ni$ ratios, respectively. The $^{208}Pb/^{206}Pb$ ratios were measured directly because interferences at these masses are negligible.

NIST Analyses using INAA: Analyses were performed in two steps [9]. Elements with short-lived irradiation products (Al, Ca, Cl, K, Mg, Mn, Na, Ti, and V) were determined by measuring duplicate 300 mg test portions from each of ten bottles of SRM 1944. The samples, standards, and controls were packaged in clean polyethylene bags and were individually irradiated for 15 s in the NIST Reactor Pneumatic Facility RT-4. Reactor power was 20 MW, which corresponds to a neutron fluence rate of about 8×10^{13} $cm^{-2} \cdot s^{-1}$. After irradiation, the samples, controls, and standards were repackaged in clean polyethylene bags and counted (gamma-ray spectrometry) three times at different decay intervals. A sample-to-detector distance (counting geometry) of 20 cm was used. Elements with long-lived irradiation products (Ag, As, Br, Co, Cr, Cs, Fe, Rb, Sb, Sc, Se, Th, and Zn) were determined by measuring one 300 mg test portion from each of nine bottles of SRM 1944. The samples, standards, controls, and blank polyethylene bags were irradiated together for a total of 1 h at a reactor power of 20 MW. Approximately four days after irradiation, the polyethylene bags were removed, and each sample, standard, control, and blank was counted at 20 cm from the detector. The samples were then recounted at 10 cm from another detector. After an additional decay time of about one month, the samples, standards, controls, and blanks were counted a third time (at 10 cm) from the second detector.

Homogeneity Assessment for Inorganic Constitutents: For some of the trace elements, most notably Cd, Fe, Pb, Rb, Sb, Sc, and Th, the variations among the test portions measured at NIST (between 0.3 g and 0.5 g) were larger than expected from the measurement process. Based on experience, it was concluded that there is some material inhomogeneity for trace elements in the test portions used. Sample variations among the NIST measurements are used as slightly conservative estimates of the sample inhomogeneities.

Particle Size Information: Dry particle-size distribution measurements for SRM 1944 were obtained as part of a collaborative effort with Honeywell's Particle and Components Measurements Laboratory (Clearwater, FL). A Microtrac particle analyzer, which makes use of light-scattering techniques, was used to measure the particle-size distribution of SRM 1944. Briefly, a reference beam is used to penetrate a field of particles and the light that scatters in the forward direction from the field is measured and the particle-size as a volume distribution is derived via a computer-assisted analysis. From these data, the total volume, average size, and a characteristic width of the particle size distribution are calculated. The system has a working range from 0.7 µm to 700 µm.

Total Organic Carbon and Percent Extractable Mass: Four laboratories provided results for total organic carbon (TOC) using similar procedures. Briefly, test portions of approximately 200 mg were reacted with 6 mol/L hydrochloric acid and rinsed with deionized water prior to combustion in a gas fusion furnace. The carbon monoxide and carbon dioxide produced were measured and compared to a blank for calculation of the percent TOC. Each laboratory analyzed test portions from six bottles of SRM 1944. For the determination of percent extractable mass, six test portions of approximately 1 g to 2 g of SRM 1944 were extracted using Soxhlet extraction for 18 h with DCM. The extraction thimbles were allowed to air dry. After reaching constant mass, the difference in the mass before and after extraction was determined.

Polychlorinated Naphthalenes: Value assignment of PCN congener concentrations was accomplished by combining results from the analysis of SRM 1944 by six laboratories that participated in an interlaboratory comparison study (see Appendix D). Each laboratory analyzed three test portions (typically 1 g to 2 g) of SRM 1944 using their routine analytical procedures that included high-resolution gas chromatography with either high-resolution mass spectrometry detection (GC-HRMS) or low-resolution MS in the negative chemical ionization mode. Calibration mixtures included either Halowax mixtures with known volume fractions of individual congeners or individual PCN congeners.

HBCDs: Value assignment of the concentrations of three HBCD isomers was accomplished by combining results from the analysis of SRM 1944 in two sets from NIST and one set from Virginia Institute of Marine Science. For the two sets analyzed at NIST, the second fraction from an acidified silica SPE clean-up was analyzed by LC/MS/MS for the HBCDs using both electrospray ionization (ESI) and atmospheric pressurized photoionization (APPI). A C18 column (3.0 mm × 150 mm × 3.5 µm column, Eclipse Plus, Agilent Technologies) and YMC Carotenoid S5 C30 column (4.6 mm × 250 mm × 5 µm column) were used with a solvent gradient using 2.5 mmol/L ammonium acetate in 12.5 % water in methanol and acetonitrile at a flow rate of 0.3 mL/min. Carbon-13 labeled HBCDs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

Table 1. Certified Mass Fraction Values for Selected PAHs in SRM 1944 (Dry-Mass Basis)

	Mass Fraction(a,b) (mg/kg)		
Phenanthrene(c,d,e,f,g)	5.27	±	0.22
Fluoranthene(c,d,e,f,g)	8.92	±	0.32
Pyrene(c,d,e,f,g)	9.70	±	0.42
Benzo[*c*]phenathrene(c,d,e,f,h)	0.76	±	0.10
Benz[*a*]anthracene(c,d,e,f,g,h)	4.72	±	0.11
Chrysene,(h,k)	4.86	±	0.10(i)
Triphenylene(h,k)	1.04	±	0.27
Benzo[*b*]fluoranthene(g,h,j)	3.87	±	0.42
Benzo[*j*]fluoranthene(h,j)	2.09	±	0.44
Benzo[*k*]fluoranthene(c,d,e,f,g,h,j)	2.30	±	0.20
Benzo[*a*]fluoranthene(c,d,e,f,h,,j)	0.78	±	0.12
Benzo[*e*]pyrene(c,d,e,f,h,j)	3.28	±	0.11
Benzo[*a*]pyrene(c,d,e,f,g,h,j)	4.30	±	0.13
Perylene(c,d,e,f,g,h,j)	1.17	±	0.24
Benzo[*ghi*]perylene(c,d,e,f,,j,k)	2.84	±	0.10
Indeno[1,2,3-*cd*]pyrene(c,d,e,f,j,k)	2.78	±	0.10
Dibenz[*a,j*]anthracene(c,d,e,f,,j,k)	0.500	±	0.044
Dibenz[*a,c*]anthracene(,j,k)	0.335	±	0.013
Dibenz[*a,h*]anthracene(j,k)	0.424	±	0.069
Pentaphene(c,d,e,f,,j,k)	0.288	±	0.026
Benzo[*b*]chrysene(c,d,e,f,j,k,h)	0.63	±	0.10
Picene(c,d,e,f,j,k)	0.518	±	0.093

(a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [10]. Each uncertainty, computed according to the Comité International des Poids et Mesures (CIPM) approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

(c) Gas chromatography/mass spectrometry (GC/MS) (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(d) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(e) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(f) GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone mixture.

(g) LC-FL of total PAH fraction after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(h) GC/MS (Sm) using a smectic liquid crystalline phase after Soxhlet extraction with DCM.

(i) The uncertainty interval for chrysene was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

(j) GC/MS (V) on 50 % phenyl-substituted methylpolysiloxane phase of extracts from GC/MS (III) and GC/MS (IV).

(k) LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

Table 2. Certified Mass Fraction Values for Selected PCB Congeners(a) in SRM 1944 (Dry-Mass Basis)

		Mass Fraction(b,c) (µg/kg)		
PCB 8	(2,4'-Dichlorobiphenyl)(d,e,f,g,h,i,j,k)	22.3	±	2.3
PCB 18	(2,2',5-Trichlorobiphenyl)(d,e,f,g,h,i,j,k)	51.0	±	2.6
PCB 28	(2,4,4'-Trichlorobiphenyl)(d,e,f,g,j,k)	80.8	±	2.7
PCB 31	(2,4',5-Trichlorobiphenyl)(d,e,f,g,j)	78.7	±	1.6(l)
PCB 44	(2,2'3,5'-Tetrachlorobiphenyl)(d,e,f,g,h,i,j,k)	60.2	±	2.0
PCB 49	(2,2'4,5'-Tetrachlorobiphenyl)(d,e,f,g,h,i,j,k)	53.0	±	1.7
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)(d,e,f,g,h,i,j,k)	79.4	±	2.0
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)(e,g,h,i,j)	71.9	±	4.3
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl)(e,g,h,i,j)	65.0	±	8.9
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl)(d,e,f,g,h,i,j)	29.9	±	4.3
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl)(d,e,f,g,h,i,j,k)	37.5	±	2.4
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl)(d,e,f,g,h,i,j,k)	73.4	±	2.5
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl)(e,f,g,h,i,j,k)	24.5	±	1.1
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl)(g,h,i,j)	63.5	±	4.7
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl)(d,e,f,g,h,i,j,k)	58.0	±	4.3
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)(d,e,f,g,h,i,j,k)	8.47	±	0.28
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)(d,e,f,g,h,i,j,k)	62.1	±	3.0
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl)(d,e,f,g,h,i,j,k)	49.7	±	1.2
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl)(d,e,f,g,h,i,j,k)	16.93	±	0.36
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)(d,e,f,g,h,i,j,k)	74.0	±	2.9
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl)(d,e,f,g,h,i,j)	6.52	±	0.66
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)(d,e,f,g,h,i,j,k)	22.6	±	1.4
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)(d,e,f,g,h,i,j,k)	44.3	±	1.2
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)(d,e,f,g,h,i,j)	12.19	±	0.57
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)(d,e,f,g,h,i,j,k)	25.1	±	1.0
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)(d,e,f,g,h,i,j)	11.2	±	1.4
PCB 195	(2,2',3,3',4,4',5,6-Octachlorbiphenyl)(d,e,f,g,h,i,j,k)	3.75	±	0.39
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)(d,e,f,g,h,i,j,k)	9.21	±	0.51
PCB 209	Decachlorobiphenyl(d,e,f,g,h,i,j,k)	6.81	±	0.33

(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [13] and later revised by Schulte and Malisch [3] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [10]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

(d) GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(e) GC-ECD (IB) on the 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

(f) GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(g) GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(h) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(i) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone mixture.

(j) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(k) Results from nineteen laboratories participating in an interlaboratory comparison exercise.

(l) The uncertainty interval for PCB 31 was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

Table 3. Certified Mass Fraction Values for Selected Chlorinated Pesticides in SRM 1944 (Dry-Mass Basis)

	Mass Fraction(a,b) (μg/kg)		
Hexachlorobenzene(e,f,g,h,i,j)	6.03	±	0.35
cis-Chlordane (α-Chlordane)(c,d,e,f,g,h,i,j)	16.51	±	0.83
trans-Nonachlor (c,d,e,f,g,h,i,j)	8.20	±	0.51

(a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [10]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

(c) GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(d) GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

(e) GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(f) GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(g) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(h) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone mixture.

(i) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(j) Results from nineteen laboratories participating in an interlaboratory comparison exercise.

Table 4. Certified Mass Fraction Values for Selected Elements in SRM 1944 (Dry-Mass Basis)

	Degrees of Freedom	Mass Fractions(a,b) (%)		
Aluminum(c,d,e)	4	5.33	±	0.49
Iron(c,d,e)	6	3.53	±	0.16
		Mass Fractions(a,b) (mg/kg)		
Arsenic(c,d,e,f,g)	10	18.9	±	2.8
Cadmium(c,f,,h,i)	6	8.8	±	1.4
Chromium(c,d,f,g,i)	9	266	±	24
Lead(c,h,i)	5	330	±	48
Manganese(c,d,e)	8	505	±	25
Nickel(c,g,h,i)	6	76.1	±	5.6
Zinc(c,d,e,g,i)	9	656	±	75

(a) The certified value is the mean of four results: (1) the mean of NIST INAA or ID-ICPMS analyses, (2) the mean of two methods performed at NRCC, and (3) the mean of results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) the mean results from INAA analyses at IAEA. The expanded uncertainty in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [11,12]. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here u_c accounts for both possible method biases, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % confidence interval with the corresponding degrees of freedom. Because of the material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.

(d) Measured at NIST using INAA.

(e) Measured at NRCC using ICPOES.

(f) Measured at NRCC using GFAAS.

(g) Measured at IAEA using INAA.

(h) Measured at NIST using ID-ICPMS.

(i) Measured at NRCC using ID-ICPMS.

Table 5. Reference Mass Fraction Values for Selected PAHs in SRM 1944

	Mass Fractions(a) (mg/kg)		
Naphthalene(b)	1.28	±	0.04(c)
1-Methylnaphthalene(b)	0.47	±	0.02(c)
2-Methylnaphthalene(b)	0.74	±	0.06(c)
Biphenyl(b)	0.25	±	0.02(c)
Acenaphthene(b)	0.39	±	0.03(c)
Fluorene(b)	0.48	±	0.04(c)
Dibenzothiophene(b)	0.50	±	0.03(c)
Anthracene(b)	1.13	±	0.07(c)
1-Methylphenanthrene(d,e,f,g)	1.7	±	0.1(h)
2-Methylphenanthrene(d,e,f,g)	1.90	±	0.06(h)
3-Methylphenanthrene(d,e,f,g)	2.1	±	0.1(h)
4-Methylphenanthrene and 9-Methylphenanthrene(d,e,f,g)	1.6	±	0.2(h)
2-Methylanthracene(d,e,f,g)	0.58	±	0.04(h)
3,5-Dimethylphenanthrene(d)	1.31	±	0.04(h)
2,6-Dimethylphenanthrene(d)	0.79	±	0.02(h,i)
2,7-Dimethylphenanthrene(d)	0.67	±	0.02(h,i)
3,9-Dimethylphenanthrene(d)	2.42	±	0.05(h,i)
1,6-, 2,9-, and 2,5-Dimethylphenanthrene(d)	1.67	±	0.03(h,i)
1,7-Dimethylphenanthrene(d)	0.62	±	0.02(h,i)
1,9- and 4,9-Dimethylphenanthrene(d)	1.20	±	0.03(h,i)
1,8-Dimethylphenanthrene(d)	0.24	±	0.01(h,i)
1,2-Dimethylphenanthrene(d)	0.28	±	0.01(h,i)
8-Methylfluoranthene(d)	0.86	±	0.02(h,i)
7-Methylfluoranthene(d)	0.69	±	0.02(h)
1-Methylfluoranthene(b)	0.39	±	0.01(c)
3-Methylfluoranthene(b)	0.56	±	0.02(c)
2-Methylpyrene(d)	1.81	±	0.04(h,i)
4-Methylpyrene(d)	1.44	±	0.03(h,i)
1-Methylpyrene(d)	1.29	±	0.03(h)
Anthanthrene(j)	0.9	±	0.1(h)

(a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) GC/MS (VI) on proprietary non-polar methylpolysiloxane phase after Soxhlet extraction with DCM.

(c) Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's t-distribution corresponding to the associated degrees of freedom (df = 2) and 95 % confidence level for each analyte.

(d) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(e) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(f) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(g) GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone mixture.

(h) The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

(i)The uncertainty interval for this compound was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

(j) LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

Table 6. Reference Mass Fractions for Selected PAHs of Relative Molecular Mass 300 and 302 in SRM 1944 (Dry-Mass Basis)

	Mass Fraction(a,b,c) (mg/kg)		
Coronene	0.53	±	0.04
Dibenzo[*b*,*e*]fluoranthene	0.076	±	0.008
Naphtho[1,2-*b*]fluoranthene	0.70	±	0.06
Naphtho[1,2-*k*]fluoranthene and Naphtho[2,3-*j*]fluoranthene	0.66	±	0.05
Naphtho[2,3-*b*]fluoranthene	0.21	±	0.01
Dibenzo[*b*,*k*]fluoranthene	0.75	±	0.06
Dibenzo[*a*,*k*]fluoranthene	0.22	±	0.02
Dibenzo[*j*,*l*]fluoranthene	0.56	±	0.03
Dibenzo[*a*,*l*]pyrene	0.12	±	0.02
Naphtho[2,3-*k*]fluoranthene	0.11	±	0.01
Naphtho[2,3-*e*]pyrene	0.33	±	0.02
Dibenzo[*a*,*e*]pyrene	0.67	±	0.05
Naphtho[2,1-*a*]pyrene	0.76	±	0.05
Dibenzo[*e*,*l*]pyrene	0.28	±	0.02
Naphtho[2,3-*a*]pyrene	0.23	±	0.01
Benzo[*b*]perylene	0.43	±	0.04
Dibenzo[*a*,*i*]pyrene	0.30	±	0.03
Dibenzo[*a*,*h*]pyrene	0.11	±	0.01

(a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's t-distribution corresponding to the associated degrees of freedom (df = 2) and 95 % confidence level for each analyte.

(c) GC/MS on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

Table 7. Reference Mass Fractions for Selected PCB Congeners(a) and Chlorinated Pesticides in SRM 1944 (Dry-Mass Basis)

	Mass Fraction(b) (µg/kg)
PCB 45 (2,2’,3,6-Tetrachlorobiphenyl)(c)	10.8 ± 1.4(d)
PCB 146 (2,2',3,4’,5,5'-Hexachlorobiphenyl)(c)	10.1 ± 1.9(d)
PCB 163 (2,3,3’,4’,5,6-Hexachlorobiphenyl)(c)	14.4 ± 2.0(d)
PCB 174 (2,2’,3,3’,4,5,6’-Heptachlorobiphenyl)(c)	16.0 ± 0.6(d)
α-HCH(f,g,h,i)	2.0 ± 0.3(e)
trans-Chlordane (γ-Chlordane)(c)	19.0 ± 1.7(d)
cis-Nonachlor(g,h,i,l,m)	3.7 ± 0.7(e)
2,4'-DDE(f,g,h,i,j,k,l,m)	19 ± 3(e)
2,4'-DDD(h,j,k,l,m)	38 ± 8(e)
4,4'-DDE(f,g,h,ihj,k,l,m)	86 ± 12(e)
4,4'-DDD (f,g,h,I,j,k,l,m)	108 ± 16(e)
4,4’-DDT(c)	170 ± 32(d)

(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [13] and later revised by Schulte and Malisch [3] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) NIST participation in the 2007 interlaboratory study using GC/MS.

(d) Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student’s t-distribution corresponding to the associated degrees of freedom (df = 2) and 95 % confidence level for each analyte.

(e) The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or he mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

(f) GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(g) GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

(h) GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(i) GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(j) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

(k) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone mixture.

(l) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts anlayzed as in GC-ECD (IIA).

(m) Results from nineteen laboratories participating in an interlaboratory comparison exercise.

Table 8. Reference Mass Fraction Values for Selected PBDEs in SRM 1944 (Dry-Mass Basis)

	Mass Fractions(a) (µg/kg)		
PBDE 47 (2,2’,4,4’-Tetrabromodiphenyl ether)(c,d,e,f)	1.72	±	0.28(b)
PBDE 99 (2,2',4,4’,5-Pentabromodiphenyl ether)(c,d,f)	1.98	±	0.26(b)
PBDE 100 (2,2',4,4’,6-Pentabromodiphenyl ether)(c,d)	0.447	±	0.027(b)
PBDE 153 (2,2',4,4’,5,5'-Hexabromodiphenyl ether)(c,d,e,f)	6.44	±	0.37(b)
PBDE 154 (2,2',4,4',5,6’-Hexabromodiphenyl ether)(c,d,f)	1.06	±	0.08(b)
PBDE 183 (2,2',3,4,4’,5',6-Heptabromodiphenyl ether)(c,d,e,f)	31.8	±	0.1(b)
PBDE 206 (2,2',3,3’,4,4',5,5',6-Nonabromodiphenyl ether)(d,e)	6.2	±	1.0(b)
PBDE 209 (Decabromodiphenyl ether) (c,d,e,f)	93.5	±	4.4(b)

(a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) Reference values are weighted means of the results from two to four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurements [11,12].

(c) Results from ten laboratories participating in an interlaboratory study for PBDEs in sediment [12].

(d) Results from four laboratories participating in the 2007 interlaboratory study [13].

(e) NIST participation in the 2007 interlaboratory study using GC/MS.

(f) Data set from NIST for PBDEs using GC/MS following PFE with alumina SPE and SEC clean-up.

Table 9. Reference Mass Fraction Values for Selected Elements in SRM 1944 (Dry-Mass Basis)

	Degrees of Freedom	Mass Fraction(a,b) (%)		
Silicon[c,d]	81	31	±	3
		Mass Fraction(a,b) (mg/kg)		
Antimony(c,e,f,g)	18	4.6	±	0.9
Beryllium(c,h)	17	1.6	±	0.3
Copper(c,d,f)	101	380	±	40
Mercury(c,i)	18	3.4	±	0.5
Selenium(c,e,f)	24	1.4	±	0.2
Silver(c,d,e,g)	8	6.4	±	1.7
Thallium(c,f)	12	0.59	±	0.1
Tin(c,f)	22	42	±	6

(a) The reference value is the equally weighted mean of available results from: (1) NIST INAA analyses, (2) two methods performed at NRCC, (3) results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) results from INAA analyses at IAEA. The expanded uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [11,12]. The value of u_c is intended to represent at the level of one standard deviation the uncertainty in the value. Here u_c accounts for possible method differences, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % confidence interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple test portions can be expected to be greater than that due to measurement variability alone.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.

(d) Measured at NRCC using GFAAS.

(e) Measured at NIST using INAA.

(f) Measured at NRCC using ID-ICPMS.

(g) Measured at IAEA using INAA.

(h) Measured at NRCC using ICPOES.

(i) Measured at NRCC using cold vapor atomic absorption spectroscopy (CVAAS).

Table 10. Reference Mass Fraction Values for Elements in SRM 1944 as Determined by INAA (Dry-Mass Basis)

	Effective Degrees of Freedom	Mass Fraction[a,b] (%)		
Calcium	21	1.0	±	0.1
Chlorine	21	1.4	±	0.2
Potassium	21	1.6	±	0.2
Sodium	25	1.9	±	0.1

	Effective Degrees of Freedom	Mass Fraction[a,b] (mg/kg)		
Bromine	10	86	±	10
Cesium	11	3.0	±	0.3
Cobalt	10	14	±	2
Rubidium	14	75	±	2
Scandium	37	10.2	±	0.2
Titanium	21	4300	±	300
Vanadium	21	100	±	9

(a) The reference value is based on the results from an INAA study. The associated uncertainty accounts for both random and systematic effects, but because only one method was used, the results should be used with caution. The expanded uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [11,12]. The value of u_c is intended to represent at the level of one standard deviation the uncertainty in the value. Here u_c accounts for possible method differences, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % confidence interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple test portions can be expected to be greater than that due to measurement variability alone.

(b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

Table 11. Reference Mass Fraction Values for
Selected Dibenzo-*p*-Dioxin and Dibenzofuran Congeners in SRM 1944 (Dry-Mass Basis)

	Mass Fraction[a,b] (µg/kg)		
2,3,7,8-Tetrachlorodibenzo-*p*-dioxin	0.133	±	0.009
1,2,3,7,8-Pentachlorodibenzo-*p*-dioxin	0.019	±	0.002
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	0.026	±	0.003
1,2,3,6,7,8-Hexachlorodibenzo-*p*-dioxin	0.056	±	0.006
1,2,3,7,8,9-Hexachlorodibenzo-*p*-dioxin	0.053	±	0.007
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin	0.80	±	0.07
Octachlorodibenzo-*p*-dioxin	5.8	±	0.7
2,3,7,8-Tetrachlorodibenzofuran[c]	0.039	±	0.015[d]
1,2,3,7,8-Pentachlorodibenzofuran	0.045	±	0.007
2,3,4,7,8-Pentachlorodibenzofuran	0.045	±	0.004
1,2,3,4,7,8-Hexachlorodibenzofuran	0.22	±	0.03
1,2,3,6,7,8-Hexachlorodibenzofuran	0.09	±	0.01
2,3,4,6,7,8-Hexachlorodibenzofuran	0.054	±	0.006[e]
1,2,3,4,6,7,8-Heptachlorodibenzofuran	1.0	±	0.1
1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.040	±	0.006[e]
Octachlorodibenzofuran	1.0	±	0.1
Total Toxic Equivalents (TEQ)[f]	0.25	±	0.01
Total Tetrachlorodibenzo-*p*-dioxins	0.25	±	0.05[e]
Total Pentachlorodibenzo-*p*-dioxins	0.19	±	0.06
Total Hexachlorodibenzo-*p*-dioxins	0.63	±	0.09
Total Heptachlorodibenzo-*p*-dioxins	1.8	±	0.2
Total Tetrachlorodibenzofurans	0.7	±	0.2
Total Pentachlorodibenzofurans	0.74	±	0.07
Total Hexachlorodibenzofurans	1.0	±	0.1
Total Heptachlorodibenzofurans	1.5	±	0.1
Total Dibenzo-*p*-dioxins[g]	8.7	±	0.9
Total Dibenzofurans[g]	5.0	±	0.5

[a] Each reference value is the mean of the results from up to fourteen laboratories participating in an interlaboratory exercise. The expanded uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [11,12] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the reference value. Here u_c is the uncertainty in the mean arising from the variation among the laboratory results. The degrees of freedom is equal to the number of available results minus one (13 unless noted otherwise). The coverage factor, k, is the value from a Student's t-distribution for a 95 % confidence interval.

[b] Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

[c] Confirmation results using a 50 % cyanopropyl phenyl polysiloxane or 90 % *bis*-cyanopropyl 10 % cyanopropylphenyl polysiloxane phase columns.

[d] Degrees of freedom = 7 for this compound.

[e] Degrees of freedom = 12 for this compound.

[f] TEQ is the sum of the products of each of the 2,3,7,8-substituted congeners multiplied by their individual toxic equivalency factors (TEFs) recommended by the North Atlantic Treaty Organization (NATO) [15]. With regard to 2,3,7,8-tetrachlorodibenzofuran, the results of the confirmation column were used when available to calculate the TEQ.

[g] Total of tetra- through octachlorinated congeners.

Table 12. Reference Values for Particle Size Characteristics for SRM 1944

Particle Measurement	Value[(a)]
Mean diameter (volume distribution, MV, µm)[(b)]	151.2 ± 0.4
Mean diameter (area distribution, µm)[(c)]	120.4 ± 0.1
Mean diameter (number distribution, µm)[(d)]	75.7 ± 0.3
Surface Area (m^2/cm^3)[(e)]	0.050 ± 0.013

[(a)] The reference value is the mean value of measurements from the analysis of test portions from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[(b)] The mean diameter of the volume distribution represents the center of gravity of the distribution and compensates for scattering efficiency and refractive index. This parameter is strongly influenced by coarse particles.

[(c)] The mean diameter of the area distribution, calculated from the volume distribution with less weighting by the presence of coarse particles than MV.

[(d)] The mean diameter of the number distribution, calculated using the volume distribution weighted to small particles.

[(e)] Calculated specific surface area assuming solid, spherical particles. This is a computation and should not be interchanged with an adsorption method of surface area determination as this value does not reflect porosity or topographical characteristics.

Table 13. Percentage of the Volume That is Smaller Than the Indicated Size

Percentile	Particle Diameter[(a)] (µm)
95	296 ± 5
90	247 ± 2
80	201 ± 1
70	174 ± 1
60	152 ± 1
50[(b)]	135 ± 1
40	120 ± 1
30	106 ± 1
20	91 ± 1
10	74 ± 1

[(a)] The reference value for particle diameter is the mean value of measurements from the analysis of test portions from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[(b)] Median diameter (50 % of the volume is less than 135 µm).

Table 14. Reference Values for Total Organic Carbon and Percent Extractable Mass in SRM 1944

	Mass Fraction (%)		
Total Organic Carbon (TOC)[a,b]	4.4	±	0.3
Extractable Mass[c,d]	1.15	±	0.04

[a] Mass fraction is reported on a dry-mass basis; material as received contains approximately 1.3 % moisture.

[b] The reference value for total organic carbon is an equally weighted mean value from routine measurements made by three laboratories. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[c] Extractable mass as determined from Soxhlet extraction using DCM.

[d] The reference value for extractable mass is the mean value of six measurements. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

Table 15. Information Mass Fraction Values for Selected Elements in SRM 1944
as Determined by INAA (Dry-Mass Basis)

	Mass Fraction[a] (%)
Magnesium[b]	1.0

	Mass Fraction[a] (mg/kg)
Cerium[b]	65
Europium[b]	1.3
Gold[b]	0.10
Lanthanum[b]	39
Thorium[b]	13
Uranium[b]	3.1

[a] Mass fraction is reported on a dry-mass basis; material as received contains approximately 1.3 % moisture.

[b] Measured at IAEA using INAA

Table 16. Information Mass Fraction Values for Selected Polychlorinated Naphthalenes in SRM 1944 (Dry-Mass Basis)

			Mass Fraction[a] (μg/kg)
PCN	19	(1,3,5-Trichloronaphthalene)	1.4
PCN	23	(1,4,5-Trichloronaphthalene)	2.4
PCN	42	(1,3,5,7-Tetrachloronaphthalene)	2.7
PCN	47	(1,4,6,7-Tetrachloronaphthalene)	3.5
PCN	52 60	(1,2,3,5,7-Pentachloronaphthalene) (1,2,4,6,7-Pentachloronaphthalene)	2.5
PCN	50	(1,2,3,4,6-Pentachloronaphthalene)	1.0
PCN	66 67	(1,2,3,4,6,7-Hexachloronaphthalene) (1,2,3,5,6,7-Hexachloronaphthalene)	0.63
PCN	69	(1,2,3,5,7,8-Hexachloronaphthalene)	1.6
PCN	73	(1,2,3,4,5,6,7-Heptachloronaphthalene)	0.51
PCN	75	(Octachloronaphthalene)	0.20

[a] Mass fractions reported on dry-mass basis; material as received contains approximately 1.3 % moisture. Information values are the median of the results from six laboratories participating in an interlaboratory comparison exercise (Appendix D).

Table 17. Information Mass Fraction Values for Three HBCD Isomers in SRM 1944 (Dry-Mass Basis)

	Mass Fraction[a,b] (μg/kg)
alpha-HBCD[b]	2.2
beta-HBCD[b]	1.0
gamma-HBCD[b]	18

[a] The information value is the median of the results from three analytical methods.
[b] Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

Table 18. Analytical Methods Used for the Measurement of Elements in SRM 1944

Elements	**Analytical Methods**
Aluminum	FAAS, ICPOES, INAA, XRF
Antimony	GFAAS, HGAAS, ICP-MS, ID-ICPMS, INAA
Arsenic	GFAAS, HGAAS, ICPMS, INAA, XRF
Beryllium	GFAAS, ICP-AES, ICPMS
Bromine	INAA
Cadmium	FAAS, GFAAS, ICPMS, ID-ICPMS
Calcium	INAA
Cerium	INAA
Cesium	INAA
Chlorine	INAA
Chromium	FAAS, GFAAS, ICPMS, ID-ICPMS, INAA, XRF
Cobalt	INAA
Copper	FAAS, GFAAS, ICPOES, ICPMS, ID-ICPMS, XRF
Europium	INAA
Gold	INAA
Iron	FAAS, ICPOES, ICPMS, ID-ICPMS, INAA, XRF
Lanthanum	INAA
Lead	FAAS, GFAAS, ICPMS, ID-ICPMS, XRF
Magnesium	INAA
Manganese	FAAS, ICPOES, ICPMS, INAA, XRF
Mercury	CVAAS, ICPMS
Nickel	GFAAS, ICPOES, ICPMS, ID-ICPMS, INAA, XRF
Potassium	INAA
Rubidium	INAA
Scandium	INAA
Selenium	GFAAS, HGAAS, ICPMS, INAA
Silicon	FAAS, ICPOES, XRF
Silver	FAAS, GFAAS, ICPMS, INAA
Sodium	INAA
Thallium	GFAAS, ICPOES, ICPMS, ID-ICPMS,
Thorium	INAA
Tin	GFAAS, ICPMS, ID-ICPMS
Titanium	INAA
Uranium	INAA
Vanadium	INAA
Zinc	FAAS, ICPOES, ICPMS, ID-ICPMS, XRF, INAA

Methods

CVAAS	Cold vapor atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
GFAAS	Graphite furnace atomic absorption spectrometry
HGAAS	Hydride generation atomic absorption spectrometry
ICPOES	Inductively coupled plasma optical emission spectrometry
ICPMS	Inductively coupled plasma mass spectrometry
ID-ICPMS	Isotope dilution inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
XRF	X-ray fluorescence spectrometry

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Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

APPENDIX A

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of PBDEs in SRM 1944 [4].

D. Hoover and C. Hamilton, AXYS Analytical, Sidney, BC, Canada
S. Klosterhaus and J. Baker, Chesapeake Biological Laboratory, Solomons, MD, USA
S. Backus, Environment Canada, Ecosystem Health Division, Burlington, ON, Canada
E. Sverko, Environment Canada, Canada Centre for Inland Waters, Burlington, ON, Canada
P. Lepom, Federal Environmental Agency, Berlin, Germany
R. Hites and L. Zhu, Indiana University, Bloomington, IN, USA
G. Jiang, Research Center for Eco-Environmental Sciences, Beijing, China
H. Takada, Tokyo University of Agriculture and Technology, Tokyo, Japan
A. Covaci and S. Vorspoels, University of Antwerp, Antwerp, Belgium
A. Li, Universtiy of Illinois at Chicago, Chicago, IL, USA

APPENDIX B

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated dibenzo-*p*-dioxins and dibenzofurans in SRM 1944.

W.J. Luksemburg, Alta Analytical Laboratory, Inc., El Dorado Hills, CA, USA
L. Phillips, AXYS Analytical Services Ltd., Sidney, British Columbia, Canada
M.J. Armbruster, Battelle Columbus Laboratories, Columbus, OH, USA
G. Reuel, Canviro Analytical Laboratories Ltd., Waterloo, Ontario, Canada
C. Brochu, Environment Québec, Laval, Québec, Canada
G. Poole, Environment Canada Environmental Technology Centre, Ottawa, Ontario, Canada
B. Henkelmann, GSF National Research Center for Environment and Health, Neuherberg, Germany
R. Anderson, Institute of Environmental Chemistry, Umeå University, Umeå, Sweden
C. Lastoria, Maxxam Analytics Inc., Mississauga, Ontario, Canada
E. Reiner, Ontario Ministry of Environment and Energy, Etobicoke, Ontario, Canada
J. Macaulay, Research and Productivity Council, Fredericton, New Brunswick, Canada
T.L. Wade, Texas A&M University, College Station, TX, USA
C. Tashiro, Wellington Laboratories, Guelph, Ontario, Canada
T.O. Tiernan, Wright State University, Dayton, OH, USA

APPENDIX C

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of trace elements in SRM 1944.

A. Abbgy, Applied Marine Research Laboratory, Old Dominion University, Norfolk, VA, USA
A. Scott, Australian Government Analytical Laboratories, Pymble, Australia
H. Mawhinney, Animal Research Institute, Queensland Department of Primary Industries, Queensland, Australia
E. Crecelius, Battelle Pacific Northwest, Sequim, WA, USA
M. Stephenson, California Department of Fish and Game, Moss Landing, CA, USA
B. Presley, Department of Oceanography, Texas A&M University, College Station, TX, USA
K. Elrick, U.S. Geological Survey, Atlanta, GA, USA

APPENDIX D

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated naphthalenes in SRM 1944.

J. Kucklick, National Institute of Standards and Technology, Charleston, SC, USA
E. Sverko, Environment Canada, Canada Centre for Inland Waters, Burlington, ON, Canada
P. Helm, Ontario Ministry of the Environment, Etobicoke, ON, Canada
N. Yamashita, National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Japan
T. Harner, Environment Canada, Meteorological Service of Canada, Toronto, ON, Canada
R. Lega, Ontario Ministry of the Environment, Etobicoke, ON, Canada