



UNITED STATES
美国
CONSUMER PRODUCT SAFETY COMMISSION
消费品安全委员会
DIRECTORATE FOR LABORATORY SCIENCES
实验室科学理事会
DIVISION OF CHEMISTRY
化学部
10901 DARNESTOWN RD
DARNESTOWN 路 10901 号
GAITHERSBURG, MD 20878
马里兰 **GAITHERSBURG** 城 20878

Test Method: CPSC-CH-C1001-09.3

测试方法: **CPSC-CH-C1001-09.3**

Standard Operating Procedure for Determination of Phthalates

邻苯二甲酸酯测定的标准操作程序

April 1st, 2010

2010 年 4 月 1 日

This document provides detailed information on test methods that will be used by the U.S. Consumer Product Safety Commission's (CPSC) testing laboratory (LSC) for the analysis of phthalate content in children's toys and child care articles covered by the standard set forth in the Consumer Product Safety Improvement Act Section 108.

这篇文章详细描述了美国消费品安全委员会（CPSC）测试室（LSC）所涵盖的消费者产品安全改进法案第 108 中规定的儿童玩具和护理品中邻苯二甲酸酯的测试方法。

It has been concluded that in order to protect children from the hazard the Act intended to address, that “concentrations of more than 0.1 percent” must apply per plasticized component part of a children's toy or child care article¹.

它决议出为保护儿童危害行为地方, 每一个儿童的玩具或儿童护理品¹的部位每种增塑剂“浓度超过 0.1%”。

¹ Statement of Policy: Testing of Component Parts with respect to Section 108 of the CPSIA, August 7, 2009 (<http://www.cpsc.gov/ABOUT/Cpsia/componenttestingpolicy.pdf>)

¹ 法律声明: 测试部件遵守消费者产品安全改进法案第 108 款, 8 月 1 日, 2009

CPSC staff has determined that using an appropriate combination of methods of extraction and analysis presented herein is sufficient to determine the concentration of the six regulated phthalates in most consumer products. Adjustments may be necessary for products made from certain materials, and should be based on sound chemistry and materials science knowledge as well as appropriate solvents for the materials. The general approach is to dissolve the sample completely in tetrahydrofuran, precipitate any PVC polymer with hexanes, filter and then dilute the solution with cyclohexane, and analyze by Gas Chromatography-Mass Spectrometry (GC-MS).

CPSC 确定用一个合适的组合方法能够测定大多数消费品中六种受限制的邻苯二甲酸酯浓度。某些特定的材料产品可能需要调整和基于健康的化学和材料科学知识也要有对材料合适的溶剂。一般通过在四氢呋喃完全溶解它，用正己烷沉淀聚氯乙烯，过滤，然后用环己烷稀释溶液，并用气相色谱质谱联用仪（GC-MS）分析。

Definitions

定义

1. Sample – An individual consumer product or a group of identical consumer products from a batch to be tested.

样品-从一批货中调出用于检测的个别消费品或一组同样的消费品。

2. Component Part – Individual sub-unit within a product.

组成部件-产品内部的子单位。

3. Laboratory Reagent Blank (LRB) – An aliquot of solvents that is treated exactly as a sample including exposure to glassware, apparatus and conditions used for a particular test, but with no added sample. LRB data are used to assess contamination from the laboratory environment.

空白实验试剂（LRB）-一种没有添加样品，同样品一样经过玻璃器皿、设备和条件下测试的一定量溶剂。LRB数据是用于评估从实验环境下受污染的程度。

4. Stock Standard– Phthalate(s) purchased from reputable commercial source at the highest available purity, used to prepare calibration standards. Replace before expiration date.

储备标准-从规范的供应商购买的用于配制校准标准纯邻苯二甲酸酯。在产品有效期前替换掉它。

5. Calibration Standard – Solutions containing the phthalate(s) of interest in cyclohexane. Each standard should contain 20µg/ml internal standard when running a 20:1 split injection, or 1µg/ml for splitless injection. A minimum of four calibration standards are used. Calibration standards should be prepared as need from the stock solution and may be stored at room temperature. Record weight of solutions before and after use to monitor for solvent evaporation. Standards should be replaced when experimental data demonstrates a decrease in quality or significant loss in solvent weight.

校准标准-在环己烷中含目标邻苯二甲酸酯浓度的溶液。当运行20:1分流进样时，每一个标准溶液必须包含20µg/mL的内标物或使用1µg/mL不分流进样。最少要用四个校准标准。校准标准如果需要，从储备溶液中重新配制并在室温下保存，记录前后溶剂的重量以监控溶剂蒸发。当超过有效期，证明质量或溶剂质量大的流失，需要替换标准溶液。

6. Quality Control Sample (QCS) – Solutions containing known amounts of phthalates that are used to evaluate the performance of the analytical instrument system. QCSs are obtained from a source external to the laboratory and are not made from the Stock Standard solutions. For example, certified reference materials (CRMs) are available from the National Institute of Standards and Technology (NIST), such as those listed in the Equipment and Supplies section below.

质量控制样（QCS）-含有用于评价分析仪器性能的已知一定量的邻苯二甲酸酯溶液。

质量控制样是从本实验室外部获得的，并非是从标准储备溶液配制得来。例如，标准物质（CRM）能从美国标准物质技术研究院得到，例如这些信息在下列仪器和供应品一节中列出。

Equipment and Supplies

仪器和供应品

The materials used for sampling and analyses are as follows:

下列这些材料用于制样和分析：

1. Tetrahydrofuran (C₄H₈O, THF), GC grade or higher
四氢呋喃（C₄H₈O, THF），色谱纯或更高
2. Hexane (C₆H₁₂), GC grade or higher
正己烷(C₆H₁₂), 色谱纯或更高
3. Cyclohexane (C₆H₁₂), GC grade or higher
环己烷(C₆H₁₂), 色谱纯或更高
4. Sealable glass vials with PTFE or silicone linere, size 20 ml or larger.
用聚四氟乙烯或硅树脂衬垫密封的20mL玻璃瓶；或更大的。

5. Cryogenic-mill (or suitable alternative to grind samples to powder)
低温研磨机（或合适可用于研磨样品至粉末的）
6. PTFE filters, 0.45 μm .
0.45 μm 聚四氟乙烯滤器
7. Gas Chromatograph-Mass Spectrometer (GC-MS) with an auto-sampler, split/splitless inlet, programmable GC oven, and capable of selective ion monitoring.
带自动进样器的气相色谱-质谱联用仪（GC-MS），分流/不分流进样，可编程的柱箱和可以进行离子选择监测。
8. CRMs containing phthalates (such as NIST SRM 3074)
含邻苯二甲酸酯的标准物质（例如NIST SRM 3074）
9. Benzyl Benzoate ($\text{C}_{14}\text{H}_{12}\text{O}_2$, BB), analytical grade or higher.
苯甲酸苄酯($\text{C}_{14}\text{H}_{12}\text{O}_2$, BB)，分析级或更高
10. Dibutyl Phthalate ($\text{C}_{16}\text{H}_{22}\text{O}_4$, DBP), CAS No. 84-74-2. analytical grade or higher.
邻苯二甲酸二丁酯($\text{C}_{16}\text{H}_{22}\text{O}_4$, DBP)，美国文摘登记号为84-74-2。分析级或更高。
11. Di-(2-ethylhexyl) phthalate ($\text{C}_{24}\text{H}_{38}\text{O}_4$, DEHP), CAS No. 117-81-7. analytical grade or higher.
邻苯二甲酸二（2-乙基）己酯($\text{C}_{24}\text{H}_{38}\text{O}_4$, DEHP), CAS No. 117-81-7.分析级或更高。
12. Benzyl Butyl Phthalate ($\text{C}_{19}\text{H}_{20}\text{O}_4$, BBP), CAS No. 85-68-7. analytical grade or higher.
邻苯二甲酸丁苄($\text{C}_{19}\text{H}_{20}\text{O}_4$, BBP), CAS No. 85-68-7. 分析级或更高
13. Di-n-octyl phthalate ($\text{C}_{24}\text{H}_{38}\text{O}_4$, DnOP), CAS No. 117-84-0. Analytical grade
邻苯二甲酸二正辛酯($\text{C}_{24}\text{H}_{38}\text{O}_4$, DnOP), CAS No. 117-84-0. 分析级或更高。
14. Diisononyl phthalate ($\text{C}_{26}\text{H}_{42}\text{O}_4$, DINP), CAS No. 28553-12-0/68515-48-0. analytical grade or higher.
邻苯二甲酸二异壬酯($\text{C}_{26}\text{H}_{42}\text{O}_4$, DINP), CAS No. 28553-12-0/68515-48-0. 分析级或更高
15. Diisodecyl phthalate ($\text{C}_{28}\text{H}_{46}\text{O}_4$, DIDP), CAS No. 26761-40-0/68515-49-1, analytical grade or higher.
邻苯二甲酸二异癸酯($\text{C}_{28}\text{H}_{46}\text{O}_4$, DIDP), CAS No. 26761-40-0/68515-49-1. 分析级

Measurement of Phthalate Concentration

邻苯二甲酸酯含量的测定

The procedure to be used for all CPSC Compliance Determinations, as described below, consists of three sections: sample preparation, extraction, and analysis. In addition to the procedure described herein, certain alternate extraction and analysis methods listed below are acceptable to CPSC staff for phthalate content certification testing. Any combination of the ten extraction and five analysis methods listed may be used. However, the CPSC staff's

Sample Preparation method must be performed prior to the alternative extraction and analysis methods.

程序依据 CPSC 规定，由如下所述的三个过程组成：样品制备、萃取和分析。在此处所述的过程外，某些供选用的萃取和分析在下表列出，这些测定邻苯二甲酸酯的方法被 CPSC 接受和证实。列出的十种萃取和五种分析方法的组合。然而在可选的萃取和分析方法前，样品制备方法必须选择 CPSC 规定的。

Sample Preparation 样品制备	Extraction Method 萃取方法	Analysis Method 分析方法
CPSC-CH-C1001-09.3	CPSC-CH-C1001-09.3	CPSC-CH-C1001-09.3
	Health Canada Method C-34 ² (http://www.hc-sc.gc.ca/cps-spc/prod-test-essai/method-chem-chim/c-34-eng.php) 加拿大安全方法 C-34 ²	
	EN 14372:2004 ³	
	EPA 3540C, Soxhlet Extraction (http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/3540c.pdf) EPA 3540C, 索氏萃取	Health Canada Method C-34
	EPA 3541, Automated Soxhlet Extraction (http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/3541.pdf) EPA 3541, 自动索氏萃取	EN 14372:2004
	EPA 3545A, Pressurized Fluid Extraction (http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/3545a.pdf) EPA 3545A, 加压流动萃取	
	EPA 3546, Microwave Extraction (http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/3546.pdf) EPA 3546, 微波萃取	EPA 8270D ⁴ (must be modified appropriately to include DINP and DIDP)
	EPA 3550C, Ultrasonic Extraction (http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/3550c.pdf) EPA 3550C, 超声萃取	EPA 8270D ⁴ (必须适当的修正 DINP 和 DIDP)
	ASTM D 2124-99 (2004) ⁵	California Dept. of Toxic Substances Control Method 加利福尼亚有毒物质控制方法
	California Dept. of Toxic Substances Control Method ⁶ 加利福尼亚有毒物质控制方法 ⁶	

Precautions

警告

These methods require the use of hazardous materials. It is paramount to properly handle all hazardous materials safely in a ventilated fume hood with adequate personal protective equipment.

这些方法要用到有危害物质。在通风橱妥善处理危险材料和佩戴适当的个人防护装备是非常重要的。

Phthalates are a common contaminant. Even low levels of contamination can impact quantitative results. Avoid plastic materials and use only scrupulously cleaned glassware and equipment. All solvents should be tested for any phthalate content. Solvent blanks should be run through the GC-MS periodically to monitor for potential contamination. Disposable glassware is recommended where practical.

邻苯二甲酸酯是一种公认的毒物。甚至低水平的污染物都能够影响定量结果。避免使用塑料材料并最好使用清洗干净了的玻璃器皿和仪器。所有溶剂必须测试所有邻苯二甲酸酯含量。定期使用空白溶剂 GC-MS 分析以监测潜在的污染物。这里推荐实用的一次性玻璃器皿。

Optional Sample Pre-Screen Using Infrared (IR) Spectroscopy

可用红外 (IR) 光谱法进行样品筛选

A broad estimate of phthalate concentration can be obtained by scanning the sample to be tested with an IR spectrometer. This information can be used when deciding the dilution factor and analysis method later in the procedure. This step is optional.

用红外光谱仪扫描可获得一种较宽范围的邻苯二甲酸酯浓度。这个信息用于决定稀释因子和这个程序的后续分析方法。这个步骤为可选。

A doublet peak at 1600 and 1580 cm^{-1} can be observed when phthalate(s) are present in amounts of ~10% or greater (see Figure 1). If this doublet is not observed, the sample should be treated as containing a low concentration of phthalates. If the doublet is present, the sample can be treated as concentrated, and a higher dilution factor may be used.

The absence of the doublet peak does **not** indicate that phthalates are not present in the sample, and that the item is compliant. Conversely, the presence of the double peak does **not** indicate that regulated phthalates are present in restricted amounts.

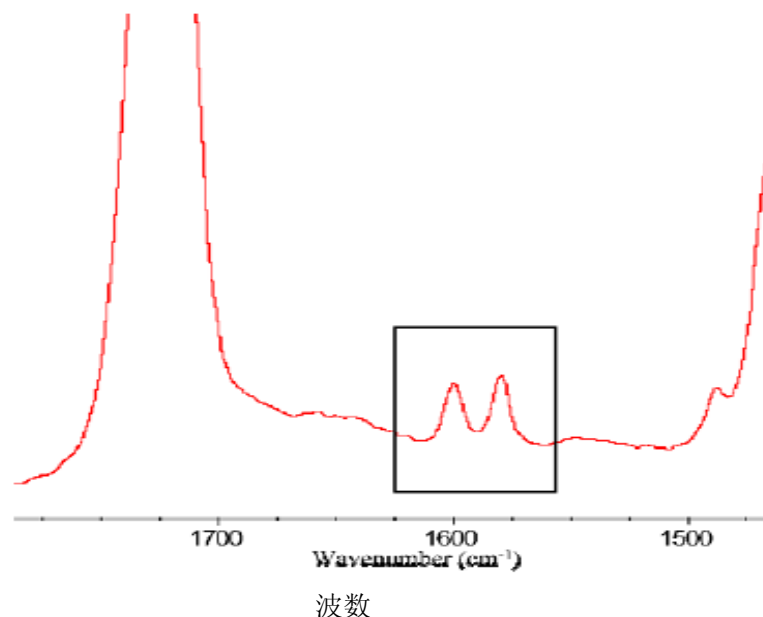
当邻苯二甲酸酯含量在约 10%或更高时注意在 1600 和 1580 cm^{-1} 处有双峰（见图 1）。如果双峰没有观察到，样品的邻苯二甲酸酯含量很低。如果双峰存在，样品浓度高，要用大的稀释因子。不存在双峰不表明样品中不含邻苯二甲酸酯和符合相关条款。相反，存在双峰不代表受控制的邻苯二甲酸酯超过限值。

² Determination of Phthalates in Polyvinyl Chloride Consumer Products

聚氯乙烯消费品中邻苯二甲酸酯的测定

Figure 1. IR spectrum of PVC plastic containing ~30% phthalates.

图 1. 聚氯乙烯塑料中含量约为 30% 的邻苯二甲酸酯的红外光谱图



Sample Preparation

样品制备

Prior to analysis, each plasticized component part should be cut into small pieces (no dimension larger than 2 mm), or milled/ground into a representative powder. Each cut/milled plasticized component part will be considered a sample for testing as described below. At minimum, prepare the amount required to constitute a sufficient sample size.

分析前，每个可塑体样品必须切成小块（尺寸不大于 2 毫米）。或碾磨/碾碎成代表性的粉末。

每份碾磨/碾碎的可塑体部件要符合下列描述才用于样品测试。制备成样品尺寸符合最小准备数量要求。

³ Child use and care articles – Cutlery and feeding utensils

儿童用品和护理用品-餐具和喂养器具

⁴ Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

气相色谱/质谱法 (GC/MS) 测半挥发性有机物

⁵ Standard Test Method for Analysis of Components in Poly(Vinyl Chloride) Compounds Using an Infrared Spectrophotometric Technique

红外分光光度计测聚氯乙烯中化合物的标准测试方法

⁶ Ting et al.; GC/MS Screening Method for Phthalate Esters in Children's Toys, Journal of AOAC

International, Vol. 92, No. 3, 2009

Ting 等人；儿童玩具中邻苯二甲酸酯的气质联用筛选方法，国际官方分析化学家协会杂志，2009 年第 3 期 92 页

Phthalate Extraction Method

邻苯二甲酸酯萃取方法

Testing lab shall determine how many replicate samples are necessary to meet their quality assurance requirements. Prepare LRB concurrently with samples.

实验室需要确定需要重复抽取样品确保它们符合质量要求。和样品同时配制空白实验试剂。

1. Weigh out a minimum of 0.05 ± 0.005 g of sample into a sealable glass vial (weighed to an accuracy of $\pm 0.5\%$ relative); if sample is not uniform, collect more to reduce sample variance.

称出最小质量为 0.05 ± 0.005 克的样品至有密封的玻璃瓶中（称量相对精确度为 $\pm 0.5\%$ ）；如果样品不一样，采集更多以较少样品离散。

2. Add 5 ml of THF to the sample. For samples larger than 0.05 g, add 10 ml of THF for every 0.1 g of sample (or a reasonable amount to dissolve sample*). Shake, stir, or otherwise mix sample for at least 30 minutes to allow dissolution*. Sample may be sonicated and/or gently heated to expedite dissolution.

加5mL四氢呋喃至样品中。如果样品大于0.05克，每0.1克样品加10mL四氢呋喃（或合理的数量以溶解样品*）。至少30分钟摇动、搅动或相反混合一次样品以使其溶解*。样品可能需要超声和/或微热以加速溶解。

*Note: Some materials may not dissolve completely. In this case, add an additional 2 hours to mixing time and then proceed.

*注：一些原料不能完全溶解。这种情况，继续混合两小时。

3. Precipitate any PVC polymer with 10 ml of hexane for every 5 ml of THF used in Step 2. Allow at least 5 minutes for polymer to settle (longer times may be necessary to minimize clogging of filters in subsequent step).

在步骤 2 中每用 5mL 四氢呋喃加 10mL 正己烷沉淀聚氯乙烯聚合物。至少让聚合体澄清 5 分钟（时间越长在后续步骤中就堵塞滤头的几率越小）。

4. (Optional) Filter THF/hexane solution through a $0.45 \mu\text{m}$ PTFE filter. Collect a few ml of filtered solution in separate vial. This step is optional, but recommended.

（可选）通过 $0.45\mu\text{m}$ 的聚四氟乙烯滤头过滤四氢呋喃/正己烷溶液。收集至少少量滤液至单独的小瓶中。这个步骤是可选项，但是推荐这样做。

5. Combine 0.3ml^* of the THF/hexane solution with 0.2ml of internal standard (BB, $150 \mu\text{g/ml}$) in a GC vial, and dilute to 1.5ml with cyclohexane.

加 0.2mL 内标物（BB, $150 \mu\text{g/mL}$ ）至 0.3mL^* 四氢呋喃/正己烷滤液中，并用环己烷稀释至 1.5mL 。

**Note:* Depending on the phthalate concentration, a different dilution ratio may be necessary to produce results in the calibration range. (e.g., if the sample is known to contain ~5% phthalate, increase the amount of filtered THF/hexane solution from 0.3 to 1.0 ml). For very low concentrations (~0.1%), use 1.3 ml of filtered solution. *A pre-screen of the sample by IR spectroscopy can provide a broad estimate of phthalate concentration.*

*注意：依据邻苯二甲酸酯的浓度，用不同的稀释比率以使结果在校准范围内。（例如，如果样品含量约为5%的邻苯二甲酸酯，增加四氢呋喃/正己烷滤液从0.3mL至1.0mL）。对于非常低的浓度（约0.1%），用1.3mL的滤液。用红外光谱法筛选获得邻苯二甲酸酯浓度的大致范围。

GC-MS Operating Procedures and Quality Control Measures

GC-MS操作程序和质量控制措施

A GC-MS system with an automatic injector is suggested for the sample analysis.

建议使用带自动进样针的 GC-MS 进行样品分析。

The following GC conditions are used (Table 1):

下列为 GC 所用条件（表 1）：

Table 1. GC Conditions

表 1.GC 条件

Column 色谱柱	DB-5MS; 30 m x .25 mm ID x 0.25 μ m
Flow Mode 流量模式	1 ml/min, constant flow (He gas) 1 ml/min, 恒流（氦气）
Inlet Mode 进样模式	20:1 Split or splitless 20: 1 分流或不分流
Injection Amount 进样量	1 μ l 1 μ L
Inlet Temperature 进样口温度	290° C 290° C
Solvent Delay 溶剂延迟	5 min 5 分钟
Initial Oven Temp, Hold Time 初始柱箱温度，保持时间	50° C, 1 min 50° C, 1 分钟
Ramp 1 程序升温 1	30° C/min, 280° C 30° C/min, 280° C
Ramp 2 程序升温 2	15° C/min, 310° C 15° C/min, 310° C
Final Hold Time 最终维持时间	4 min or longer 4 分钟或更长

The 20:1 split mode injection should be used when the phthalate concentration is expected to be $\geq 5\%$. All other samples are run in splitless mode. *A pre-screen of the sample by IR spectroscopy can provide a broad estimate of phthalate concentration.*

当邻苯二甲酸酯浓度大于期望值的5%时需要进行20:1分流进样模式。其它所有样品使用不分流模式。用红外光谱法筛选获得邻苯二甲酸酯浓度的大致范围。

Samples are analyzed using both full scan mode and the Selective Ion Monitoring (SIM) program listed in Table 2. Monitor for corresponding ions of each compound listed in a time segment (e.g., set Group 3 to monitor for 149, 167, 261, 279, 293, and 307 m/z). The retention times listed are based on CPSC data, and must be confirmed by analyzing stock standards. The last column indicates the identification (ID) ion, and the relative abundance of this ion to 149 m/z .

样品分析用全扫描模式和选择离子监测模式程序列于下表2。在特定的阶段扫描每个所列表中混合物中对应的离子（例如，设置SIM第3阶段扫描149、167、261、279、293和307 m/z ）。保留时间基于 CPSC 的数据，必须用分析储备标准物质来确认。最后一栏显示确认（ID）离子和 149 m/z 这个离子的相对丰度。

If the instrument to be used has limited SIM abilities, monitor for only those ions in **bold**.

如果所用仪器的SIM能力有限，仅需监视用**粗体**标识的离子。

Table 2. SIM Settings

表2.选择离子监测设置

	Estimated Retention Time (min) 估计相对时间（分钟）	Corresponding Ions (m/z) 对应离子	Published Relative Abundance of ID Ion to 149 m/z⁷ 发布的149确认离子相对丰度
SIM Group 1: 第一组离子监测	5 - 9.5 Minutes 5 - 9.5分钟		
BB (Internal Standard) 苯甲酸苄酯（内标物）	7.9	91.1, 105 , 194, 212	
DBP	8.5	149, 167, 205, 223	223: 4
SIM Group 2: 第二组离子监测	9.5-10.8 Minutes 9.5-10.8分钟		
BBP	9.8	91.1, 149, 206	206: 27
DEHP	10.4	149, 167, 279	279: 10
SIM Group 3: 第三组离子监测	10.8 – End		
DnOP	11.2	149, 167, 261, 279	279: 12
DINP	11.6	149, 167, 293	293: 26
DIDP	12.1	149, 167, 307	307: 27

Analysis

分析

1. Prepare at least four calibration standards for each of the six phthalates of interest along with one calibration blank (cyclohexane). Each calibration standard should have an internal standard concentration of 20 µg/ml (for 20:1 split mode samples) or 1 µg/ml (for splitless mode samples).

配制至少含6个目标分析物的邻苯二甲酸酯的四个校准标准连同一个校准空白（环己烷）。每个校准标准必须含20µg/mL的内标物（20:1分流比模式样品）或1µg/mL（不分流模式样品）。

2. Analyze standards and blank with the GC-MS in both full-scan mode and SIM. Qualitatively analyze the results to ensure proper retention times and no contamination.

用GC-MS全扫描模式和选择离子监测模式分析标准溶液和空白溶液。定量分析结果以确定合适的保留时间和没有污染物。

3. Integrate the peak area from valley to valley (approximate retention times are listed in Table 2) for each standard. Compounds monitored in SIM Groups 1 and 2 can be quantified by extracted ion chromatograph (EIC) or the ion chromatograph (suggested quantitative ions are in **bold**). The phthalates scanned in SIM Group 3 overlap and **must** be quantified using their quantitative ions (again, in **bold**).

对每个标准从峰谷到峰谷的峰面积进行积分（近似的保留时间范围列于表2中）。在通过提取离子色谱图中第1组和第2组的SIM监测化合物或离子色谱图（建议用**粗体**的定量离子）进行积分。在第3组SIM中邻苯二甲酸酯重叠，必须用它的定量离子积分（再次，用**粗体**）。

4. Construct a calibration curve using normalized phthalate responses. The normalize phthalate response (Pht_n) is calculated by:

用邻苯二甲酸酯标准信号创建校准曲线。计算邻苯二甲酸酯的响应因子(Pht_n):

$$Pht_n = \frac{Pht}{ISTD}$$

Where Pht is the phthalate response and $ISTD$ is the internal standard response.

这里 Pht 是邻苯二甲酸酯的响应值， $ISTD$ 是内标物的响应值。

⁷ Bolgar, M; Hubball, J; Groeger, J; Meronek, S; *Handbook for the Chemical Analysis of Plastic and Polymer Additives*, CRC Press, Boca Raton, FL, 2008.

奥加尔,M;鲍尔,J; Meronek, S;塑料和聚合物添加剂化学分析手册,CRC出版社, Boca Raton, FL, 2008.

5. Analyze a CRM to ensure a proper calibration. The analyzed value should be within $\pm 15\%$ of the expected value. If not, prepare new standards and re-run calibration.

分析CRM以确定合适的校准浓度。分析值应在预期值的 $\pm 15\%$ 之内。如果不在，配制新的标准溶液，然后重新校正。

6. Analyze the LRB and all samples.

分析试剂空白溶液和所有样品。

7. Qualitatively evaluate full-scan results. Phthalates of interest should be identified by matching with retention times and mass spectra of standards. Potential non-regulated chemicals which may have mass ions of interest and/or similar retention times and must be qualitatively eliminated from consideration based on their spectra and chromatograms include, but are not limited to, linear C9 and C10 phthalates, and terephthalates.

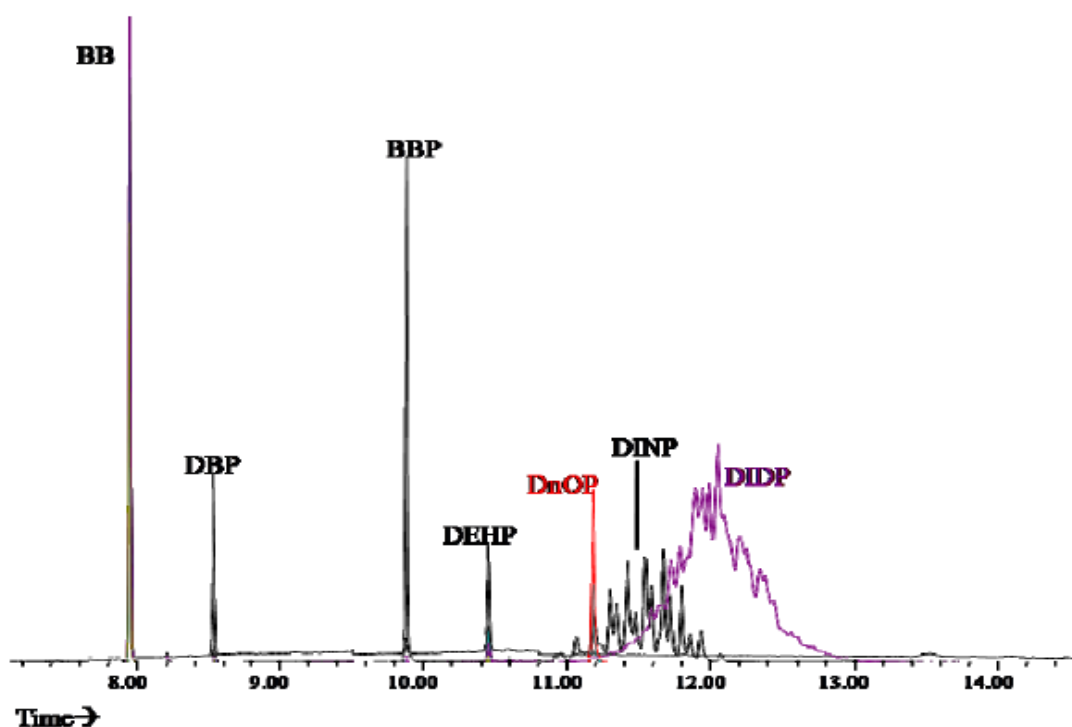
评估全扫描定量结果。通过保留时间和标准质谱图确认目标邻苯二甲酸酯。潜在的非控制化学品有目标质量离子和/或相似的保留时间，必须定量的从它们的质谱和包括色谱中扣除，但是不限于含9个碳和10个碳的直线型邻苯二甲酸酯和对苯二甲酸酯。

8. Quantitate SIM results. If the results are out of the calibration range, return to step 5 of the phthalate extraction method (perform another dilution to get results in calibration range). If signal is near or below the limit of detection, change the inlet mode to splitless injection (when using splitless injection, samples and calibration curves should contain an internal standard concentration of $1 \mu\text{g/ml}$). If signal is still below quantitation limits when using a splitless injection, the injection amount can be doubled from $1 \mu\text{l}$ to $2 \mu\text{l}$ (calculations must be adjusted accordingly).

SIM结果定量。如果结果超出校准范围，重复邻苯二甲酸酯方法步骤5（另外稀释使结果落入校准范围内）。如果信号接近或低于检出限，更改为不分流进样模式（当用不分流进样，样品和校准曲线溶液需含浓度为 $1 \mu\text{g/mL}$ 的内标物）。当用不分流进样如果信号仍然低于检出限，进样体积从 $1 \mu\text{L}$ 加倍成 $2 \mu\text{L}$ （从而校准必须调整）。

Figure 2. Chromatogram overlay of all phthalates of interest and internal standard.

图 2.所有目标邻苯二甲酸酯和内标物的色谱图。



Calculations and Results

计算结果

Results can be reported as follows:

结果按下面示例报告:

$$\text{Percentage [Phthalate]} = \% \text{ Phthalate (w/w)} = [(C \times V \times D) / (W \times 1000)] \times 100$$

$$\text{百分比[邻苯二甲酸酯]} = \% \text{ 邻苯二甲酸酯 (w/w)} = [(C \times V \times D) / (W \times 1000)] \times 100$$

Where

这里

C = Concentration of phthalate in GC-MS sample (in $\mu\text{g/ml}$)

C=GC/MS样品中邻苯二甲酸酯的浓度（用 $\mu\text{g/mL}$ 表示）

V = Total volume of THF and hexanes added from steps 2 and 3 of phthalate extraction method

V=邻苯二甲酸酯萃取方法步骤2和3中加入四氢呋喃和正己烷的总体积

D = Dilution factor from step 5 of phthalate extraction method

D=邻苯二甲酸酯萃取方法步骤5中的稀释因子

W = Weight of sample collected (in mg)

W=收集到的样品重量（用mg表示）

Repeat calculation for each phthalate present in sample

重复计算样品中出现的每个邻苯二甲酸酯含量

Example:

例如:

A small, homogeneous PVC toy was cut into small pieces and ground to a powder. 50 mg of sample powder was dissolved in 5 ml THF; next 10 ml of hexane were added (total of 15 ml of solvent). Of the filtered solution, 0.3 ml was combined with 0.2 ml of internal standard and diluted with cyclohexane to 1.5 ml for GC-MS analysis (5 times dilution factor). The GC-MS results found 200µg/ml of DEHP and 50µg/ml of DINP. Therefore, the sample contained 30% DEHP and 7.5% DINP by weight.

小的，同类聚氯乙烯玩具切成小片和制成粉末。50mg 样品溶于 5mL 四氢呋喃中；接下来加入 10mL 正己烷（溶剂总体积为 15mL）。加 0.3mL 滤液至 0.2mL 内标物中及用环己烷稀释至 1.5mL 用于 GC-MS 分析（5 倍稀释因子）。GC-MS 结果显示 200µg/mLDEHP 和 50µg/mL DINP。因此，样品含 DEHP 和 DINP 质量分数分别为 30%和 7.5%。

W	C	V	D	$[(C \times V \times D) / (W \times 1000)] \times 100$
Sample weight 样品重量	Measured DINP Concentration by GC-MS	Original Volume 原始体积	Dilution Factor 稀释因子	% DINP (w/w)
50 mg	200 µg/ml	15 ml	1.5 ml / 0.3 ml = 5	$[(200 \text{ µg/ml} \times 15 \text{ ml} \times 5) / (50 \text{ mg} \times 1000 \text{ µg/mg})] \times 100\% = \mathbf{30\%}$
	Measured DINP Concentration by GC-MS GC-MS 测试 DINP 浓度			% DIDP (w/w)
	50 µg/ml			$[(50 \text{ µg/ml} \times 15 \text{ ml} \times 5) / (50 \text{ mg} \times 1000 \text{ µg/mg})] \times 100\% = \mathbf{7.5\%}$