**UNITED STATES**
**CONSUMER PRODUCT SAFETY COMMISSION**
**4330 EAST WEST HIGHWAY**
**BETHESDA, MD 20814**

This document has been electronically approved and signed.

**DATE:** January 17, 2018

**BALLOT VOTE SHEET**

**TO:** The Commission
Alberta E. Mills, Acting Secretary

**THROUGH:** Patricia M. Hanz, General Counsel
Patricia H. Adkins, Executive Director

**FROM:** Patricia M. Pollitzer, Assistant General Counsel
David M. DiMatteo, Attorney, OGC

**SUBJECT:** Draft final rule: *Revision to the Notice of Requirements for Prohibitions of Children's Toys and Child Care Articles Containing Specified Phthalates*
Ballot Vote Due - Tuesday, January 23, 2018

Staff is forwarding to the Commission a memorandum recommending that the Commission issue a final rule to revise the existing notice of requirements (NOR) for the prohibitions on phthalates in children's toys and child care articles to make the NOR consistent with a final phthalates rule published in the *Federal Register* on October 27, 2017. The Office of the General Counsel is providing for the Commission's consideration the attached draft final rule for publication in the *Federal Register*.

Please indicate your vote on the following options:

I. Approve publication of the attached document in the *Federal Register*, as drafted.

_____ _____
(Signature) (Date)

II. Approve publication of the attached document in the *Federal Register*, with changes. (Please specify.)

_____ _____
(Signature) (Date)

III. Do not approve publication of the attached document in the *Federal Register*.

_____

_____

_____

_____

_____ _____

(Signature) (Date)

IV. Take other action. (Please specify.)

_____

_____

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_____ _____

(Signature) (Date)

Attachment: Draft *Federal Register* Final rule: *Revision to the Notice of Requirements for Prohibitions of Children's Toys and Child Care Articles Containing Specified Phthalates*

[Billing Code 6355-01-P]

CONSUMER PRODUCT SAFETY COMMISSION

**[Docket No. CPSC-2017-0043]**

**16 CFR Part 1112**

**CPSC Acceptance of Third Party Laboratories: Revision to the Notice of Requirements for Prohibitions of Children's Toys and Child Care Articles Containing Specified Phthalates**

**AGENCY**: Consumer Product Safety Commission.

**ACTION**: Final rule.

**SUMMARY**: This final rule updates the existing notice of requirements (NOR) for prohibitions of children's toys and child care articles containing specified phthalates that provide the criteria and process for Commission acceptance of accreditation under the Consumer Product Safety Act (CPSA). The final NOR revises the current NOR to be consistent with the regulated phthalates in children's toys and child care articles in the final phthalates rule published in the *Federal Register* on October 27, 2017.

**DATES:** This rule will become effective on April 25, 2018. The incorporation by reference of the publication listed in this rule is approved by the Director of the Federal Register, as of April 25, 2018.

**FOR FURTHER INFORMATION CONTACT**: Scott R. Heh, Project Manager, Directorate for Laboratory Sciences, Consumer Product Safety Commission, 5 Research Place, Rockville, MD 20850; telephone: 301-504-7646; email: sheh@cpsc.gov.

**SUPPLEMENTARY INFORMATION:**

## A. Background

Section 108 of the Consumer Product Safety Improvement Act of 2008 (CPSIA) established requirements concerning concentration limits for specified phthalates in children's toys and child care articles. In accordance with section 108 of the CPSIA, on October 27, 2017, the Commission published a final phthalates rule in the *Federal Register*. 82 FR 49938. The final rule makes permanent the interim prohibition on children's toys that can be placed in a child's mouth and child care articles that contain concentrations of more than 0.1 percent of diisononyl phthalate (DINP). The phthalates rule extends this prohibition to cover all children's toys and child care articles containing concentrations of more than 0.1 percent of DINP. The phthalates rule also lifts the interim prohibitions on children's toys that can be placed in a child's mouth and child care articles that contain concentrations of more than 0.1 percent of di-n-octyl phthalate (DNOP) or diisodecyl phthalate (DIDP). In addition, the phthalates rule prohibits children's toys and child care articles that contain concentrations of more than 0.1 percent of diisobutyl phthalate (DIBP), Di-n-pentyl phthalate (DPENP), di-n-hexyl phthalate (DHEXP), and dicyclohexyl phthalate (DCHP). The permanent prohibitions on children's toys and child care articles that contain concentrations of more than 0.1 percent on the use of di-(2-ethylhexyl) phthalate (DEHP), dibutyl phthalate (DBP), and benzyl butyl phthalate (BBP) in children's toys and child care articles in section 108 of the CPSIA are unchanged by the phthalates rule.

On October 27, 2017, in the same issue of the *Federal Register*, the Commission published a notice of proposed rulemaking (NPR) to update the existing notice of

requirements (NOR) for prohibitions of children's toys and child care articles containing specified phthalates. As explained further below, NORs provide the criteria and process for Commission acceptance of accreditation of third party testing laboratories that test products' conformance to CPSC requirements. The Commission had previously issued an NOR for the statutory phthalate provisions. The October 27, 2017 NPR proposed to revise the NOR to reflect the phthalates prohibited in children's toys and child care articles in the final phthalates rule. Because the phthalates rule revises the list of statutorily prohibited phthalates in children's toys and child care articles in section 108 of the CPSIA, this final rule amends the existing NOR for the prohibitions of children's toys and child care articles containing specified phthalates so that the NOR will reflect those changes.

**B. Notice of Requirements**

Section 14(a) of the CPSA requires that products subject to a consumer product safety rule under the CPSA, or to a similar rule, ban, standard, or regulation under any other act enforced by the Commission, be certified as complying with all applicable CPSC requirements. 15 U.S.C. 2063(a). Such certification must be based on a test of each product, or on a reasonable testing program. Products that are subject to a children's product safety rule must be certified based on tests of a sufficient number of samples by a third party conformity assessment body accredited by the Commission to test according to the applicable requirements. The Commission's phthalates rule is considered a "children's product safety rule." 15 U.S.C. 2063(f). Thus, products subject to the phthalates rule are subject to the testing and certification requirements of section 14 of the CPSA.

Because children's toys and child care articles are children's products, samples of these products must be tested by a third party conformity assessment body whose accreditation has been accepted by the Commission. These products also must comply with all other applicable CPSC requirements, such as the lead content requirements of section 101 of the CPSIA, the requirements of the toy standard, 16 CFR part 1250, and the tracking label requirement in section 14(a)(5) of the CPSA.

In accordance with section 14(a)(3)(B)(vi) of the CPSIA, the Commission has previously published two NORs for accreditation of third party conformity assessment bodies for testing children's toys and child care articles under section 108 of the CPSIA (76 FR 49286 (Aug. 10, 2011), 78 FR 15836 (March 12, 2013)).

As described in the NPR, the Commission will use the following process during the transition period from test method CPSC-CH-C1001-09.3 (2010) to a revised version of the method, test method CPSC-CH-C1001-09.4 (2018). CPSC will accept testing to support children's toys and child care article certifications to the new phthalates prohibitions if the laboratory is already CPSC-accepted to test to CPSC-CH-C1001-09.3 (2010). Laboratories that conduct testing to support product certifications to the new phthalates prohibitions must list in their test reports "16 CFR part 1307" and CPSC-CH-C1001-09.3 until laboratories have transitioned their accreditation scope and CPSC listing to CPSC-CH-C1001-09.4 (2018).

The CPSC will open the laboratory application process for test method CPSC-CH-C1001-09.4 (2018) on the date the final NOR rule is published in the *Federal Register*. Laboratories that seek CPSC acceptance to the revised prohibitions for children's toys and child care articles in 16 CFR part 1307 will be required to update their

accreditation scope. To be CPSC-accepted, a laboratory's scope of accreditation must include the reference to CPSC-CH-C1001-09.4 (2018). Laboratories that are currently CPSC-accepted to CPSC-CH-C1001-09.3 (2010) are instructed to update their accreditation scope to include CPSC-CH-C1001-09.4 (2018) as soon as possible, and submit their application for CPSC acceptance. Laboratories that were not previously CPSC-accepted to CPSC-CH-C1001-09.3 (2010) are instructed to work with their accreditation bodies to include "CPSC-CH-C1001-09.4 (2018)" in their scope documents.

CPSC will accept testing results to the new phthalates prohibitions in 16 CFR part 1307 from laboratories that are CPSC-accepted to CPSC-CH-C1001-09.3 (2010) for two years from the date of publication of the final rule NOR in the *Federal Register*. This should allow adequate time for laboratories to work with their accreditation bodies to make official updates to their accreditation scope document to include the revised CPSC method "CPSC-CH-C1001-09.4 (2018)" and submit applications to the CPSC. On [I**nsert date 2 years after publication in the FEDERAL REGISTER**], the CPSC will no longer accept laboratory applications that reference CPSC-CH-C1001-09.3 (2010), and any application to CPSC must reference "CPSC-CH-C1001-09.4 (2018)."

### C. Comments on the NPR

We received four comments on the NPR. Three comments addressed the DRAFT CPSC procedure CPSC-CH-C1001-09.4(2017) that was published with the October 2017 NPR briefing package. The first comment requested clarification of the final list of prohibited phthalates. The second comment highlighted "that dissolved PVC-samples can be precipitated by adding hexane.

The phthalates remain in solution. The centrifuged solution can then be measured in the GC." The third comment came from a testing laboratory representative who recommended a few changes to add clarity and more specificity to the CPSC procedure. The fourth comment was outside the scope of the rule.

Staff made editorial clarifications to the DRAFT CPSC procedure based on the comments. Staff revised the test procedure to clarify the final list of eight prohibited phthalates. Also, staff made several additions to the test equipment and supplies section of the test method reflected in test method CPSC-CH-C1001-09.4 (January 2018) in response to comment.

Staff did not accept some of the commenters' suggested changes to the test method. The revised test method does not add a temperature specification to the sonication reference in the extraction steps because the extraction is not heat dependent. Additionally, the revised test method does not include suggested additional elements to the Table 1 Conditions for Gas Chromatography-Mass Spectrometry (GC-MS). Staff did not make changes to Table 1, as well as other recommended quality assurance changes to the analysis section of the test method, in order to allow accredited laboratories flexibility in setting up their internal standard operating and quality assurance procedures. Adding the suggested requirements to Table 1 might have forced accredited laboratories to alter already suitable quality assurance programs, thus reducing flexibility. The comment relating to use of hexane for PVC samples did not warrant a change to the test method because the test method already permits the use of hexane.

**D. Description of the Rule**

The final rule amends 16 CFR part 1112(b)(31), (b)(31)(i), and (c)(i) to update the references to reflect the promulgation of 16 CFR part 1307 and revised CPSC test method CPSC-CH-C1001-09.4 (2018). CPSC test method CPSC-CH-C1001-09.4 (2018), has, among other things, been updated to reflect the list of phthalates prohibited in children's toys and child care articles in 16 CFR part 1307 (DEHP, DBP, BBP, DNOP, DIBP, DPENP, DHEXP, or DCHP). CPSC test method CPSC-CH-C1001-09.4 (2018) provides detailed information on the test methods that will be used by the CPSC testing laboratory for the analysis of phthalate content in children's toys and child care articles covered by the standard set forth in section 108 of the CPSIA and 16 CFR part 1307. The test method provides detailed information regarding equipment and supplies, the procedure for the measurement of phthalate concentration, sample preparation, the phthalate extraction method, and instrument parameters. The test method CPSC-CH-C1001-09.4 (2018) is substantially the same as the current testing procedure.

**E. Incorporation by Reference**

The Office of the Federal Register (OFR) has regulations concerning incorporation by reference. 1 CFR part 51. Under these regulations, agencies must discuss, in the preamble to the final rule, ways that the materials the agency incorporates by reference are reasonably available to interested persons and how interested parties can obtain the materials. In addition, the preamble to the final rule must summarize the material. 1 CFR 51.5(b).

In accordance with the OFR's requirements, section D of this preamble summarizes CPSC test method CPSC-CH-C1001-09.4 (2018) that the Commission

incorporates by reference into 16 CFR part 1112. The test method is reasonably available to interested parties, and interested parties may obtain a copy of the test method from CPSC National Product Testing and Evaluation Center, 5 Research Place, Rockville, MD 20850; www.cpsc.gov. The test method will also be available on the CPSC website. https://cpsc.gov/Business--Manufacturing/Testing-Certification/Lab-Accreditation/Test-Methods/. A copy of the test method can also be inspected at CPSC's Office of the Secretary, U.S. Consumer Product Safety Commission, Room 820, 4330 East West Highway, Bethesda, MD 20814, telephone 301-504-7923.

**F. Effective Date**

The APA generally requires that a substantive rule must be published not less than 30 days before its effective date. 5 U.S.C. 553(d)(1). The NPR proposed a 30-day effective date because the rule allows testing to continue under the existing testing method by testing laboratories that meet certain criteria for a period of up to two years after the publication of a final rule. However, to avoid possible confusion if the NOR effective date differed from the effective date for the underlying phthalates rule, we are setting the effective date for the rule on April 25, 2018, the same date the phthalates rule takes effect. This is consistent with past practice setting the effective date for NORs for durable nursery products under section 104 and updates to the mandatory toy standard ASTM F963 on the same date the underlying rule takes effect.

**G. Regulatory Flexibility Act**

The Regulatory Flexibility Act (RFA) requires an agency to prepare a regulatory flexibility analysis for any rule subject to notice and comment rulemaking requirements under the APA, or any other statute, unless the agency certifies that the rulemaking will

not have a significant economic impact on a substantial number of small entities. 5 U.S.C. 603 and 605. Small entities include small businesses, small organizations, and small governmental jurisdictions.

The Commission certified, in the NPR, that the rule would not have a significant impact on a substantial number of small entities because the revised testing method is substantially the same as the method that laboratories are already using, qualified testing laboratories should be able to adopt the new method without difficulty, and the 2-year window allowed to amend the accreditation scope documents would allow testing laboratories to time the amendments with their periodic reassessments by their accreditation bodies, which should result in minimal (if any) additional cost. The Commission did not receive any public comments that addressed the potential impact on small entities, nor has the Commission staff become aware of any new information that would change its previous determination regarding the impact on small entities.

**H. Environmental Considerations**

The Commission's regulations provide a categorical exclusion for the Commission's rules from any requirement to prepare an environmental assessment or an environmental impact statement because they "have little or no potential for affecting the human environment." 16 CFR 1021.5(c)(2). This rule falls within the categorical exclusion, so no environmental assessment or environmental impact statement is required.

**List of Subjects in 16 CFR Part 1112**

Administrative practice and procedure, Audit, Consumer protection, Incorporation by reference, Reporting and recordkeeping requirements, Third party conformity assessment body.

For the reasons discussed in the preamble, the Commission amends Title 16 CFR chapter II, as follows:

**PART 1112—REQUIREMENTS PERTAINING TO THIRD PARTY CONFORMITY ASSESSMENT BODIES**

1. The authority citation for part 1112 continues to read as follows:

**Authority:** 15 U.S.C. 2063; Pub. L. 110–314, section 3, 122 Stat. 3016, 3017 (2008).

2. Amend § 1112.15 by:

a. Revising the introductory text to paragraph (b)(31);

b. Revising paragraph (b)(31)(i); and

c. Revising paragraph (c)(3)(i).

The revisions read as follows:

**§ 1112.15 When can a third party conformity assessment body apply for CPSC acceptance for a particular CPSC rule or test method?**

* * * * *

(b) * * *

(31) 16 CFR part 1307, Prohibition of Children's Toys and Child Care Articles Containing Specified Phthalates. For its accreditation to be accepted by the Commission to test for phthalates in children's toys and child care articles, a third party conformity

assessment body must have one or more of the following test methods referenced in its statement of scope:

(i) CPSC Test Method CPSC-CH-C1001-09.4, "Standard Operating Procedure for Determination of Phthalates";

* * * * *

(c) * * *

(3) * * *

(i) CPSC-CH-C1001-9.4, "Standard Operating Procedure for Determination of Phthalates", January 17, 2018.

* * * * *

Dated: _____

_____
Alberta E. Mills,
Acting Secretary
U.S. Consumer Product Safety Commission

# Staff Briefing Package

# Final Rule:
# Amendment to the Notice of Requirements for 16 C.F.R. Part 1112 for CPSC Acceptance of Third Party Laboratories for Prohibited Phthalates in Children's Toys and Child Care Articles

January 17, 2018

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**BRIEFING MEMORANDUM**

**UNITED STATES**
**CONSUMER PRODUCT SAFETY COMMISSION**
**4330 EAST WEST HIGHWAY**
**BETHESDA, MD 20814**

**Memorandum**

This document has been electronically approved and signed.

January 17, 2018

TO : The Commission
Alberta E. Mills, Acting Secretary

THROUGH: Patricia M. Hanz, General Counsel
Patricia H. Adkins, Executive Director
DeWane Ray, Deputy Executive Director for Safety Operations

FROM : George A. Borlase, Ph.D., P.E., Assistant Executive Director
Office of Hazard Identification and Reduction
Scott R. Heh, Program Manager, Third Party Laboratory Accreditation
Directorate for Laboratory Sciences

SUBJECT : Final Rule: Amendment to the Notice of Requirements in 16 C.F.R. part 1112 for CPSC Acceptance of Third Party Laboratories for Prohibited Phthalates in Children's Toys and Child Care Articles

# I. Introduction

Section 14 of the Consumer Product Safety Act (CPSA) requires manufacturers of children's products to certify that their products comply with all applicable children's product safety rules. Certification must be based on third party testing. Section 14(a)(3) of the CPSA requires the Commission to publish a notice of requirements (NOR) for the accreditation of third party testing laboratories to assess whether a children's product conforms to the applicable children's product safety rule. The Commission's requirements for CPSC acceptance of third party testing laboratories are found in 16 C.F.R. part 1112. Part 1112 states the criteria for laboratory acceptance and certification timing for the determination of prohibited phthalates in children's toys and child care articles that section 108 of the Consumer Product Safety Improvement Act of 2008 (CPSIA) established.

Section 108 of the CPSIA establishes concentration limits for specified phthalates in children's toys and child care articles. Under section 108 of the CPSIA the Commission issued a final rule on October 27, 2017 modifying some of the phthalate restrictions previously in place under the CPSIA. 82 FR 49938.

The Commission's phthalates final rule made permanent the interim prohibition on children's toys that can be placed in a child's mouth and child care articles that contain concentrations of more than 0.1 percent of diisononyl phthalate (DINP). The phthalates rule extended this prohibition to cover all children's toys and child care articles

containing concentrations of more than 0.1 percent of DINP. The phthalates rule also lifted the interim prohibitions on children's toys that can be placed in a child's mouth and child care articles that contain concentrations of more than 0.1 percent of di-n-octyl phthalate (DNOP) or diisodecyl phthalate (DIDP). In addition, the phthalates rule prohibited children's toys and child care articles that contain concentrations of more than 0.1 percent of diisobutyl phthalate (DIBP), Di-n-pentyl phthalate (DPENP), di-n-hexyl phthalate (DHEXP), and dicyclohexyl phthalate (DCHP). The permanent prohibitions on children's toys and child care articles that contain concentrations of more than 0.1 percent on the use of di-(2-ethylhexyl) phthalate (DEHP), dibutyl phthalate (DBP), and benzyl butyl phthalate (BBP) in children's toys and child care articles in section 108 of the CPSIA were unchanged by the phthalates rule. The rule at 16 C.F.R. part 1307 will become effective on April 25, 2018.

On October 27, 2017, the Commission published a notice of proposed rulemaking (NPR) to revise the current NOR to reflect the changes to the prohibitions of children's toys and child care articles containing specified phthalates and to allow for third party testing laboratories to transition to an updated CPSC testing method to determine whether the specified phthalates are present in children's toys and child care articles. 82 FR 49767. The comment period on the NPR closed on January 10, 2018.

This memorandum presents staff's recommendation for a final rule revising the existing NOR found in part 1112 for CPSC acceptance of accreditation of testing laboratories so that the NOR reflects the revised phthalates prohibitions at 16 C.F.R. part 1307. The recommendation for the NOR also addresses the transition to an updated version of "Test Method: CPSC-CH-C1001-09 Standard Operating Procedure for Determination of Phthalates." The version number for the CPSC test method will be updated from CPSC-CH-C1001-09.3 to CPSC-CH-C1001-09.4.

## II. Laboratory Competence and CPSC Acceptance of Accreditation

The regulation at 16 C.F.R. part 1112 establishes a baseline requirement that, to be considered for CPSC acceptance, a laboratory must be accredited to International Organization for Standardization (ISO)/International Electrotechnical Commission (IEC) Standard ISO/IEC 17025:2005, "General Requirements for the Competence of Testing and Calibration Laboratories" (ISO/IEC 17025). The laboratory's accreditation body must be a signatory to the International Laboratory Accreditation Cooperation – Mutual Recognition Arrangement (ILAC-MRA). Laboratories that are ISO/IEC 17025 accredited have been assessed to have the technical and managerial competence to conduct testing in accordance with the standards and test methods that are listed in the laboratory's scope of accreditation. The scope of accreditation is issued and made publically available by the laboratory's accreditation body.

Currently, the CPSC accepts the accreditation of more than 400 laboratories to conduct testing in accordance with the CPSC procedure, "CPSC-CH-C1001-09.3 - Standard Operating Procedure for Determination of Phthalates, April 1st, 2010." CPSC staff developed the procedure to support the first NOR for phthalates in children's toys and child care articles prohibited under the CPSIA.

CPSC staff in the Laboratory Sciences Directorate, Division of Chemistry (LSC), developed a revised and updated version of the CPSC Standard Operating Procedure for Determination of Phthalates, CPSC-CH-C1001-09.4 (2018) [Tab A]. The revised testing procedure is substantially the same as the current version. The revision reflects changes in the list of prohibited phthalates and includes updates to ASTM and other standards that are referenced within the CPSC method.

CPSC staff considers testing laboratories that have been CPSC-accepted for CPSC-CH-C1001-09.3 to have the competency to conduct testing to a newly issued and revised CPSC method, CPSC-CH-C1001-09.4, given the limited differences between the two.

## III. Acceptance of Accreditation and Third Party Testing to Support Certification to the Revised Phthalates Prohibition

Staff recommends that the Commission approve an approach for acceptance of testing that allows certification to the revised prohibitions of children's toys and child care articles containing specified phthalates, and acceptance of laboratory accreditation that takes into account testing laboratories that are currently CPSC-accepted for testing in accordance with CPSC-CH-C1001-09.3 (2010).

Staff also recommends the CPSC accept testing results to the new phthalates rule at 16 C.F.R. part 1307 by laboratories that are CPSC-accepted to CPSC-CH-C1001-09.3 (2010) for a period not to exceed 2 years after publication of the final NOR. This allows adequate time for testing laboratories to work with their accreditation bodies to update their official accreditation scope document to include the revised CPSC method, "CPSC-CH-C1001-09.4 (2018)," and submit applications to the CPSC.

The CPSC will open the laboratory application process for CPSC-CH-C1001-09.4 (2018) when a final rule NOR is published in the *Federal Register* as an amendment to 16 C.F.R. part 1112. Testing laboratories that seek CPSC acceptance to the revised prohibitions for children's toys and child care articles will be required to update their accreditation scope. To be CPSC-accepted, a laboratory's scope of accreditation must include the reference to CPSC-CH-C1001-09.4 (2018). Laboratories that are currently CPSC-accepted to CPSC-CH-C1001-09.3 (2010) will be instructed to update their accreditation scope to include CPSC-CH-C1001-09.4 (2018) as soon as possible, and submit their application for CPSC acceptance. Laboratories that were not previously CPSC-accepted to CPSC-CH-C1001-09.3 (2010) and request CPSC acceptance to CPSC-CH-C1001-09.4 (2018) will be instructed to work with their accreditation bodies to include "CPSC-CH-C1001-09.4 (2018)" in their scope documents.

Beginning 2 years after the date of publication of the final rule NOR, the CPSC will no longer accept laboratory applications that reference CPSC-CH-C1001-09.3 (2010). At that time, the scope document submitted with applications to CPSC must reference "CPSC-CH-C1001-09.4 (2018)."

The CPSC will provide notice of these requirements through publication of the final rule in the *Federal Register* and through direct email to all current CPSC-accepted laboratories and their accreditation bodies.

This approach should allow for a practicable transition from CPSC-CH-C1001-09.3 (2010) to CPSC-CH-C1001-09.4 (2018) for testing laboratories, children's products manufacturers, and other interested parties.

## IV. NPR Comments

Four comments were submitted on the NPR [Docket ID: CPSC-2017-0043 on Regulations.gov]. Three comments were on the DRAFT CPSC procedure CPSC-CH-C1001-09.4 that was published with the October 2017 NPR briefing package. The first commenter requested clarification of the final list of prohibited phthalates. The second commenter highlighted "that dissolved PVC-samples can be precipitated by adding hexane. The phthalates remain in solution. The centrifuged solution can then be measured in the GC." The third commenter is a testing laboratory representative who recommended a few changes to add clarity and more specificity to the CPSC procedure. The fourth comment was outside the scope of the rule.

Staff made editorial clarifications to the DRAFT CPSC procedure based on the comments. Staff revised the test procedure to clarify the final list of eight prohibited phthalates. Also, staff made several additions to the test equipment and supplies section of the test method CPSC-CH-C1001-09.4 (January 2018) [Tab A].

Staff did not accept some of the commenters' suggested changes to the test method. The revised test method does not add a temperature specification to the sonication reference in the extraction steps because the extraction is not heat dependent. Additionally, the revised test method does not include suggested additional elements to the Table 1 Conditions for Gas Chromatography-Mass Spectrometry (GC-MS). Staff did not make changes to Table 1, as well as other recommended quality assurance changes in the analysis section, in order to allow accredited laboratories flexibility in setting up their internal standard operating and quality assurance procedures. Adding the suggested requirements to Table 1 might have forced accredited laboratories to alter already suitable quality assurance programs, thus reducing flexibility. The comment relating to use of hexane for PVC samples did not warrant a change to the test method because the test method already permits the use of hexane.

## V. Impact on Small Businesses (Regulatory Flexibility Act)

In accordance with the requirements of the Regulatory Flexibility Act, staff analyzed the impact of the proposed rule on small entities, including small businesses. Staff concluded the impact of the proposed rule on small testing laboratories would be minimal because the revised testing method is similar to the method that laboratories are already using and qualified testing laboratories should be able to adopt the new method without difficulty. Additionally, the 2-year window allowed by the proposed rule to amend the accreditation scope documents would allow testing laboratories to time the amendments with their periodic reassessments by their accreditation bodies, which should result in minimal additional cost (if any). Based on this analysis, the Commission certified in the NPR that the proposed rule would not have a significant impact on a substantial number of small entities. The Commission did not receive any public comments that addressed

the potential impact on small entities, nor has Commission staff discovered any new information that would suggest that the rule could have a larger impact on small entities. Therefore, the certification that the rule would not have a significant impact on a substantial number of small entities is still appropriate.

## VI. Recommendation

Staff recommends that the Commission publish the draft final rule amending the NOR in 16 C.F.R. part 1112. The Office of the General Counsel is providing a draft *Federal Register* notice for the final rule.

The NPR proposed a 30-day effective date because the rule allows testing to continue under the existing testing method by testing laboratories that meet certain criteria for a period of up to two years after the publication of a final rule. We received no comments regarding the effective date. However, to avoid possible confusion if the NOR effective date differed from the effective date for the underlying phthalates rule, we are setting the effective date for the rule on April 25, 2018, the same date the phthalates rule takes effect. This is consistent with past practice setting the effective date for NORs for durable nursery products under section 104 and updates to the mandatory toy standard ASTM F963 on the same date the underlying rule takes effect.

## TAB A - Test Method: CPSC-CH-C1001-09.4 Standard Operating Procedure for Determination of Phthalates

**UNITED STATES**
**CONSUMER PRODUCT SAFETY COMMISSION**
**DIRECTORATE FOR LABORATORY SCIENCES**
**DIVISION OF CHEMISTRY**
**5 RESEARCH PLACE**
**ROCKVILLE, MD 20850**

# Test Method: CPSC-CH-C1001-09.4
# Standard Operating Procedure for Determination of Phthalates
**January 17, 2018**

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 analyzing phthalate content in children's toys and child care articles covered by the standard set forth in section 108 of the Consumer Product Safety Improvement Act Section and 16 C.F.R. part 1307. In 2017, the Commission at 16 C.F.R. part 1307 issued a rule prohibiting children's toys and child care articles containing specified phthalates. Guidance regarding the law can be found at: https://www.cpsc.gov/en/Business--Manufacturing/Business-Education/Business-Guidance/Phthalates-Information.

CPSC staff has determined that using an appropriate combination of the methods of extraction and analysis presented here is sufficient to determine the concentration of the eight 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 dissolved polymer with a second solvent, then analyze by Gas Chromatography-Mass Spectrometry (GC-MS).

## Definitions

1. Sample – An individual consumer product or a group of identical consumer products from a batch to be tested.
2. Component Part – Individual sub-unit within a product.
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.
4. Stock Standard – Phthalate(s) purchased from reputable commercial source at the highest available purity, used to prepare calibration standards. These must be replaced before expiration date.
5. Calibration Standard – Solutions containing the phthalate(s) of interest. Each standard should contain 20 µg/mL of internal standard. A minimum of four calibration standards

are used. Calibration standards should be prepared, as needed, from the stock solution and may be stored at room temperature. Standards should be replaced when experimental data demonstrates a decrease in quality.

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.

**Equipment and Supplies**

The materials used for sampling and analyses are as follows:

**1.** Tetrahydrofuran ($C_4H_8O$, THF), GC grade or higher.
**2.** Hexane ($C_6H_{14}$), GC grade or higher.
**3.** Acetonitrile ($C_2H_3N$), GC grade or higher.
**4.** Sealable glass vials with PTFE or silicone liner, size 20 mL or larger.
**5.** Analytical balance, capable of weighing to ±0.0001 g.
**6.** Cryogenic-mill (or suitable alternative to grind samples to powder).
**7.** Ultrasonic Bath.
**8.** PTFE filters, 0.45 μm.
**9.** Gas Chromatograph-Mass Spectrometer (GC-MS) with an auto-sampler, split/splitless inlet, programmable GC oven, and capable of selective ion monitoring.
**10.** GC vials, size 2 mL.
**11.** Volumetric glassware
**12.** Volumetric pipettes
**13.** CRMs containing phthalates (such as NIST SRM 2860 or Korea Research Institute of Standards and Science CRM 113-03-006).
**14.** Benzyl Benzoate ($C_{14}H_{12}O_2$, BB), analytical grade or higher.
**15.** Dibutyl Phthalate ($C_{16}H_{22}O_4$, DBP), CAS No. 84-74-2, analytical grade or higher.
**16.** Diisobutyl phthalate ($C_{16}H_{22}O_4$, DIBP), CAS No. 84-69-5, analytical grade or higher.
**17.** Di-n-pentyl phthalate ($C_{18}H_{26}O_4$, DPENP), CAS No. 131-18-0, analytical grade or higher.
**18.** Di-n-hexyl phthalate ($C_{20}H_{30}O_4$, DHEXP), CAS No. 84-75-3, analytical grade or higher.
**19.** Dicyclohexyl phthalate ($C_{20}H_{26}O_4$, DCHP), CAS No. 84-61-7, analytical grade or higher.
**20.** Di(2-ethylhexyl) phthalate ($C_{24}H_{38}O_4$, DEHP), CAS No. 117-81-7, analytical grade or higher.
**21.** Benzyl Butyl Phthalate ($C_{19}H_{20}O_4$, BBP), CAS No. 85-68-7, analytical grade or higher.
**22.** DINP ($C_{26}H_{42}O_4$):
- 1,2-Benzenedicarboxylicacid, 1,2-diisononyl; CAS No. 28553-12-0, analytical grade or higher.

-1,2-Benzenedicarboxylicacid, di-$C_{8-10}$ branched alkyl esters, $C_9$-rich; CAS No. 68515-48-0, technical grade.

**Measurement of Phthalate Concentration**
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 here, certain alternative methods listed below are acceptable to CPSC staff for phthalate content certification testing. Note that some methods require adaptation to be sufficient. Most notably, some methods do not specifically include all of the phthalates that are regulated in use in children's toys and child care items in the United States. As such, the methods must be modified to ensure including all phthalates of interest.

*Standalone methods*: These methods are acceptable to CPSC staff for phthalate content certification testing, as written, and they require no adaptations beyond including all phthalates of interest:
- CPSC-CH-C1001-09.4 and CPSC-CH-C1001-09.3
- Health Canada Method C34 and C34.2
- EN 14372:2004
- EN 71-5:1993/A1:2006
- ASTM D7823-16
- ASTM D8133-17
- GB/T 22048-2015
- ISO 8124-6 2014
- ISO 14389:2014 (*for textiles only*)
- California Department of Toxic Substances Control Method[1]

*Extraction only*: The following methods are only suitable for the extraction portion of certification testing. They must be combined with an analysis method from a standalone method listed above, or an *analysis only* method listed in the following section.
- EPA 3540C, 3541, 3545A, 3546 and 3550C
- ASTM D2124-99 (2011)

*Analysis only*: The following methods must be combined with a sample preparation and extraction method from a standalone method or extraction only method.
- EPA 8270D (must be modified appropriately to include all phthalates of interest).

*References:* The following standard guide is recommended to assist users interpreting results.
- ASTM D7993-15.

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

[1] Ting et al.; GC/MS Screening Method for Phthalate Esters in Children's Toys, Journal of AOAC International, Vol. 92, No. 3, 2009.

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.

**Sample Preparation**

Before 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.

**Phthalate Extraction Method**

Testing lab shall determine how many replicate samples are necessary to meet their quality assurance requirements. Prepare LRB concurrently with samples. Add benzyl benzoate directly to the precipitation solvent (choice of acetonitrile or hexane) to yield a concentration of 30 µg/mL for use as an internal standard.

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

**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 to expedite dissolution.

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

**3.** Precipitate any PVC polymer with 10 mL of acetonitrile or hexane for every 5 mL of THF used in Step 2. Shake vigorously and allow at least 5 minutes for polymer to settle.

**4.** Transfer supernatant solution to GC vial for analysis. If hexane was used for Step 3, it is recommended to filter the supernatant solution through a 0.45 µm PTFE filter before transferring.

**Instrument Parameters**

A GC-MS system with an auto-sampler is suggested for the sample analysis. Related instrumentation, such as GC or liquid chromatography (LC) with advanced MS options (*e.g.*, ion trap or tandem mass spectrometry) can be used for qualitative assessment. The following GC conditions are used (Table 1):

**Table 1.** GC Conditions

| | |
|---|---|
| Column | DB-5MS; 30 m x 0.25 mm ID x 0.25 µm |
| Flow Mode | 1 mL/min, constant flow (He or $H_2$ gas) |
| Inlet Mode | 20:1 Split |
| Injection Amount | 1 µl |
| Inlet Temperature | 290° C |
| Solvent Delay | 4.5 minutes |
| Initial Oven Temp, Hold Time | 150° C, 1 min |
| Ramp 1 | 30° C/min, 280° C |
| Ramp 2 | 15° C/min, 310° C |
| Final Hold Time | 3 minutes or longer |

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, 249, 279, and 293 *m/z*). The retention times listed are based on CPSC data using helium gas, 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*. An example chromatogram is shown in Figure 1.

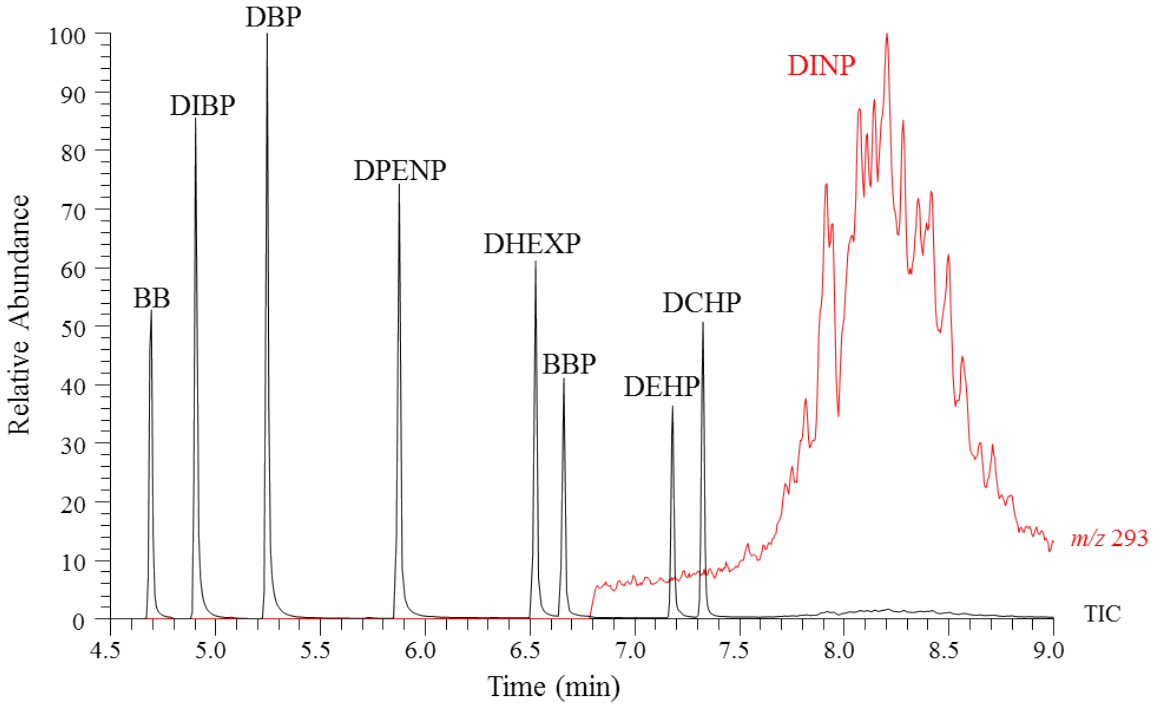
If the instrument to be used has limited SIM abilities, monitor for only those ions in **bold**.

**Table 2.** SIM Settings

| | ***Estimated* Retention Time (min)** | **Corresponding Ions (*m/z*)** | **Published Relative Abundance of ID Ion to 149 *m/z*[2]** |
|---|---|---|---|
| *SIM Group 1:* | *4.5 – 5.3 minutes* | | |
| BB (Internal Standard) | 4.69 | 91.1, **105**, 194, 212 | |
| DIBP | 4.91 | 149, 167, 205, **223** | **223**: 9.6 |
| DBP | 5.25 | 149, 167, 205, **223** | **223**: 4 |
| *SIM Group 2:* | *5.5 – 7.0 minutes* | | |
| DPENP | 5.88 | 149, 219, **237** | **237**: 6.1 |
| DHEXP | 6.53 | 149, 233, **251** | **251**: 4.5 |
| BBP | 6.66 | 91.1, 149, **206** | **206**: 27 |
| *SIM Group 3:* | *7.0 – End* | | |
| DEHP | 7.18 | 149, 167, **279** | **279**: 32 |
| DCHP | 7.33 | 149, 167, **249** | **249**: 4.5 |
| DINP | 7.8-8.9 | 149, 167, **293** | **293**: 26 |

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

**Figure 1**. Total ion chromatogram (TIC) of the internal standard and all phthalates of interest, overlaid with an extracted ion chromatogram isolating DINP (red, *m/z* 293).




## Analysis

**1.** Prepare at least four calibration standards for each of the eight phthalates of interest along with one calibration blank. Each calibration standard should have an internal standard concentration of 20 µg/mL.

**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.

**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 monitored in SIM Group 3 overlap and **must** be quantified using their quantitative ions (again, in **bold**).

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

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

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

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

**6.** Analyze the LRB and all samples. Analyze a CRM if time has passed since the last calibration check.

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

**8.** Quantitate SIM results. If the results are out of the calibration range, prepare additional calibration standards or dilute the sample for re-analysis.

**Calculations and Results**

Results can be reported as follows:

Percentage [Phthalate] = % Phthalate (w/w) = [(C x V) / (W x 1000)] x 100

Where

C = Concentration of phthalate in GC-MS sample (in µg/mL)

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

W = Weight of sample collected (in mg)

Repeat calculation for each phthalate present in sample

**Example**

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 acetonitrile were added (total of 15 mL of solvent). The GC-MS results found 20 µg/mL of DEHP and 5 µg/mL of DINP. Therefore, the sample contained 0.60% DEHP and 0.15% DINP by weight.

<table>
<tr><td>W</td><td>C</td><td>V</td><td>[(C x V) / (W x 1000)] x 100</td></tr>
<tr><td>Sample weight</td><td>Measured DEHP Concentration by GC-MS</td><td>Original Volume</td><td>% DEHP (w/w)</td></tr>
<tr><td rowspan="3">50 mg</td><td>20 µg/mL</td><td rowspan="3">15 mL</td><td>[(20 µg/mL x 15 mL) / (50 mg x 1000 µg/mg)] x 100% = 0.60%</td></tr>
<tr><td>Measured DINP Concentration by GC-MS</td><td>% DINP (w/w)</td></tr>
<tr><td>5 µg/mL</td><td>[(5 µg/mL x 15 mL) / (50 mg x 1000 µg/mg)] x 100% = 0.15%</td></tr>
</table>

**Summary of Changes in Revision CPSC-CH-C1001-09.4**

-Page 1; added link to CPSC website for guidance.
-Page 2; updated list to reflect currently prohibited phthalates; additional clarity provided for the definition of DINP.
-Page 3; updated acceptable alternative methods section to improve clarity and include new options, including reference section.
-Page 4; removed optional IR pre-screen due to modification of test method.
-Page 4; added option to use acetonitrile instead of hexane. Acetonitrile was determined to yield a better precipitation, eliminating the need for filtering the solution before analysis.
-Page 4; internal standard is now directly added to precipitation solvent prior to sample preparation.
-Page 4; alternative technical options are listed under **Instrument Parameters**.
-Page 4; new GC parameters are listed in **Table 1** to decrease analysis time.
-Page 5; **Table 2** is updated with new retention times to reflect the updated GC parameters and current prohibited phthalates.
-Page 6; **Figure 1** was updated to reflect the updated GC parameters and current prohibited phthalates.
-Page 7; Example calculation updated to reflect modification of test method.