Designation: F1313 – 90 (Reapproved 2011)

Standard Specification for Volatile *N*-Nitrosamine Levels in Rubber Nipples on Pacifiers[1]

This standard is issued under the fixed designation F1313; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification applies to the nitrosamine content of rubber used in the manufacture of nipples for infant pacifiers.

1.2 This specification does not apply to plastic nipples (on pacifiers).

1.3 The purpose of this specification is to establish a maximum level of allowed nitrosamines in rubber nipples and to outline a uniform testing method to determine such level.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.5 The following precautionary statement pertains only to the test method portions, Sections 5, and Appendix X4 of this specification. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards are given in Appendix X2.

2. Terminology

2.1 *Definitions:*

2.1.1 *lot*—normal production run or, in the case of imports, a shipment of items produced in the same time frame.

2.1.2 *nitrosamines*—chemically active compounds principally formed by the reaction of amines with oxides of nitrogen present in the environment.

3. Significance and Use

3.1 This specification is intended for use in reducing the normal exposure to nitrosamines.

3.2 This specification refers only by way of example to the eight volatile *N*-nitrosamines identified below:

3.2.1 *N*-nitrosodimethylamine,
3.2.2 *N*-nitrosodiethylamine,
3.2.3 *N*-nitrosodibutylamine,
3.2.4 *N*-nitrosomorpholine,
3.2.5 *N*-nitrosopiperidine,
3.2.6 *N*-nitrosopyrrolidine,
3.2.7 *N*-ethylphenylnitrosamine.

4. Test Method

4.1 Determine nitrosamine levels by using either the methylene chloride extraction method described in the collaborative study conducted by the National Center for Toxicological Research[2] or the Food and Drug Administration method.[2]

5. Acceptable Level

5.1 A test sample of nipples, drawn from a standard production lot, shall not contain more than 10 ppb (in each of 3 aliquots) of any one nitrosamine. In addition, the total nitrosamines of the sample shall not exceed 20 ppb.

5.2 Each manufacturer or distributor of the product shall test the product in such a manner and at such intervals to ensure compliance in accordance with the methodology prescribed by the test procedure utilized. Records of all testing shall be retained for a period of up to three years.

6. Report

6.1 Report the following information:

6.1.1 Lot number,
6.1.2 Date samples,
6.1.3 Date tested,
6.1.4 Individual nitrosamine content, and
6.1.5 Total nitrosamine content.

[1] This specification is under the jurisdiction of ASTM Committee F15 on Consumer Products and is the direct responsibility of Subcommittee F15.22 on Toy Safety.

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[2] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

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APPENDIXES

(Nonmandatory Information)

X1. BACKGROUND

X1.1 This specification provides the rationale for the drafting of a voluntary product standard establishing acceptable levels and testing procedures for nitrosamines contained in children's rubber pacifiers.

X1.2 Some nitrosamines are known to be potent animal carcinogens and are suspected human carcinogens. In 1981, the West German Government enacted regulations limiting the amount of preformed nitrosamine in rubber pacifiers. Nitrosamines are formed from amines used as accelerators during vulcanization of the rubber or are unintentional trace substances present in stabilizers used in the manufacturing process.

X1.3 In 1982, the Consumer Product Safety Commission (CPSC) began meeting with rubber pacifier manufacturers and importers (most are imported), drawing their attention to both the carcinogenic potential as measured by laboratory bioassays on rodents and the results of an audit of those pacifiers on the market. The audit revealed nitrosamine levels ranging from "non-detectable" to as much as hundreds of parts per billion (ppb). The Toy Manufacturers of America (TMA) undertook to coordinate a program to lower the levels of nitrosamines and validate a single test method that could be duplicated in laboratories worldwide. This effort was a joint, round-robin program with the CPSC, the National Center for Toxicological Research (NCTR) and pacifier manufacturers/importers. Another method of testing has been detailed by the Food and Drug Administration in their program to reduce nitrosamine levels in nursing nipples.

X1.4 This specification currently recognizes two test methods, one developed by the National Center for Toxicological Research (NCTR) (see Appendix X3), and one which is known as the Food and Drug Administration (FDA) method (see Appendix X4). Both methods have been corroborated and adopted as an approved method by the Association of Official Analytical Chemists. The process by which these methods were corroborated and adopted ensures that the methods are reproducible both within and between laboratories and that the methods provide equivalent test results. Several government and independent laboratories participated in the corroborative study in which coded quadruplicate samples of three composites were sent to each laboratory for analysis and tally, conclusively providing evidence of reproducibility among laboratories.

X1.5 The Consumer Product Safety Commission uses the NCTR method in analyzing pacifiers for nitrosamine content under its enforcement policy.[3] The FDA utilizes the FDA method in its Compliance Policy Guide, 7117.15.[4] The CPSC and NCTR staffs characterize the NCTR method as cheaper, faster, and more reproducible, although both the NCTR and FDA have affirmed that their two methods give essentially the same results in their laboratories.

X1.6 The test methodologies contained in Appendix X3 and Appendix X4 define sample sizes and contain the requisite and prescribed procedure for sampling from a lot to be tested.

X1.7 On December 27, 1983, the CPSC issued a statement of policy that rubber pacifiers are hazardous substances as defined in Section 2(g) of the Federal Hazardous Substances Act and are banned if they contain more than 60 ppb of nitrosamines as measured by the NCTR methylene chloride extraction test, effective January 1, 1984.

X1.8 A collaborative study between the NCTR, manufacturers/importers and leading testing laboratories was initiated to validate the test for consistent results between laboratories. Manufacturers and importers have continued to work with manufacturing processes and independent laboratories to reduce nitrosamine levels during this period. Significant progress has been made since the start of the program.

X1.9 In June, 1985, a group of manufacturers met with the Toy Manufacturers of America to draft a voluntary specification. That specification was presented to a task force of consumers and manufacturers on August 14, 1985 at ASTM Headquarters. This specification is the result of the corrections and suggestions made at that meeting, as well as comments from formal ASTM balloting procedures.

[3] Federal Register 48, No. 249, pp. 56988–56990, available from Superintendent of Documents, U.S. Government Printing Office, North Capitol and H Streets, NW, Washington, DC 20401.

[4] Federal Register 49, No. 252, pp. 50789–50790, available from Superintendent of Documents, U.S. Government Printing Office, North Capitol and H Streets, NW, Washington, DC 20401.

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X2. HAZARD ANALYSIS

X2.1 The scientific community in Europe, Canada and the United States has concluded that nitrosamines are suspected human carcinogens. However, the actual risk to infants who use rubber pacifiers is probably very small. In fact, a risk assessment study conducted by the Rubber Manufacturers Association involving infant feeding nipples concluded on a worst case basis that the lifetime risk to a user of infant nipples (having 60 ppb nitrosamines) was one in 23 million. However, the Toy Manufacturers Association has approached this problem, accepting that high levels of nitrosamines are unacceptable and that low levels of 20 ppb, that generally represent unavoidable contamination, are achievable.

X3. PROCEDURE FOR ANALYSIS OF *N* -NITROSAMINES IN PACIFIERS—A COLLABORATIVE STUDY

X3.1 *Reagents, Apparatus, and Pacifiers*—All solvents were distilled in glass and all other reagents were chemically pure grade.

X3.1.1 *N-Nitrosamine Standard Stock:*

X3.1.1.1 *External Standard Stock*—Ten µg/mL in ethanol of 7 *N*-nitrosamine mixture.

X3.1.1.2 *Internal Standard Stock*—A solution of NDPA (5 µg/mL in ethanol).

X3.1.2 *Pacifiers.*

X3.1.3 *Mineral Oil*— White, light weight Saybolt viscosity 125/135.

X3.1.4 *Nitrosation Inhibitor*—Ten mg alpha-Tocopherol/mL mineral oil.

X3.1.5 *Keeper Solution:*

X3.1.5.1 *For K-D Evaporation*—Eighty mg mineral oil/mL dichloromethane.

X3.1.5.2 *For N_2 Blowdown*—Twenty mg mineral oil/mL iso-octane.

X3.1.6 *ThermoSorb/N*[7] *Cartridges*—Used as received for quantitative trapping of volatile *N*-nitrosamines.

X3.1.7 *Variable Temperature Oil Bath*—Thermostatically controlled oil bath capable of operating at 150 ± 3°C and of moving vertically with aid of a lab jack.

X3.1.8 *Purge and Trap Apparatus*—The apparatus shown in Fig. X3.1 contains the following parts:

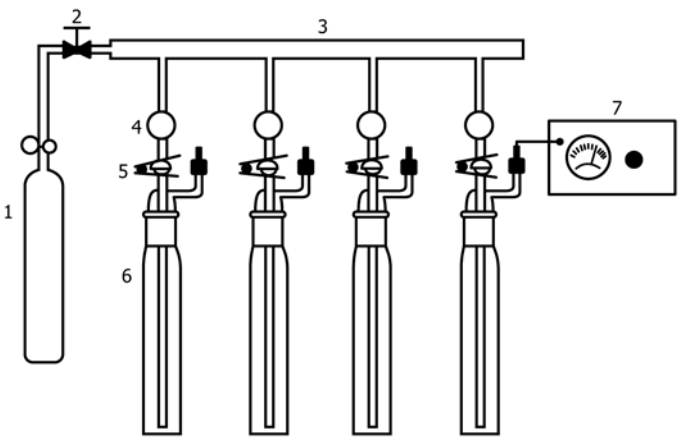

FIG. X3.1 Diagram of Purge and Trap Apparatus Equipped With Four Impinger Tubes

X3.1.8.1 Argon (Ar) gas cylinder and gage;

X3.1.8.2 Metering valve;

X3.1.8.3 Purge gas manifold 4-position;

X3.1.8.4 Nalgene needle valve Type CPE (No. 6400-0125);

X3.1.8.5 Ground glass outer joints with pinch clamps, 18/7;

X3.1.8.6 Impingers, 50 mL graduated glass tubes with 24/40 clear-seal grease free joints, 18/7 ground glass ball joints, and 1 mm inside diameter nozzle approximately 5 mm above the bottom of the impinger; and

X3.1.8.7 *Variable Scale Flow-Check*—Calibrated for purge rate in mL/min, of argon. A bubble meter for measuring gas flow rates for a gas chromatograph may be substituted.

NOTE X3.1—Do not use any rubber tubing, gaskets, o-rings, or any other items made of rubber in any part of this method.

X3.2 *Description and Use of the Purge and Trap Apparatus*—The apparatus shown in Fig. X3.1 was designed for the high temperature purging and trapping of seven volatile nitrosamines from a concentrated sample extract/mineral oil mixture on four samples simultaneously. A cylinder containing prepurified argon (Ar) gas equipped with a high pressure regulator was used to supply 20 psig to a flow metering valve that regulates the final purge flow through the samples. The gas stream was diverted into a tubular stainless steel manifold 250 by 20 mm outside diameter containing four exit tubes spaced 50 mm apart and measuring 40 by 10 mm outside diameter. Each of these tubes were coupled using 9.52 mm (⅜ in.) Tygon tubing to Nalgene needle valves that serve dual purposes: as a shut off valve when assaying less than four samples; and for making minor adjustments in purge rate due to slight differences in flow characteristics of the impinger and ThermoSorb/N cartridges. An 18/7 ground glass outer spherical joint was attached to the Nalgene valve to permit a quick, gas tight connection to the 18/7 ground glass ball joint on the impinger inlet using the appropriate pinch clamp. As shown in Fig. X3.2 the impingers were assembled by inserting the glass nozzle (1 mm inside diameter orifice) into the sample mixture and coupling the 24/40 grease free male and female joints together forming a leak free seal. Once sealed, the Ar gas was allowed to purge through the sample mixture, through the outlet tube of the impinger (see Fig. X3.2). Tygon tubing was used to connect the impinger outlet tube to the inlet side (marked "AIR IN") of the ThermoSorb/N cartridge, that is simply a standard male luer syringe connector. The purged volatile *N*-nitrosamines were then collected on the sorbent

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