Percent phthalates determination[*]

Only those specimens consisting of PVC plasticized with DINP or DEHP were quantitatively analyzed for the amount of plasticizer present. Approximately 0.05 g of PVC was dissolved in 5 ml of tetrahydrofuran (THF) at room temperature. The PVC polymer was then precipitated with 10 ml of hexane and filtered through a Pall Gelman GHP Acrodisc 0.45 μm filter. A 50 μL aliquot of the THF/hexane solution and 20 μg (80 μg of 250 μg/mL) of the internal standard (benzyl butyl phthalate, BBP)[†] were diluted to 20 ml of cyclohexane for quantitative determination by GC/MS.

Analytical determination

Infrared (IR) - the IR spectrum was obtained using a Nicolet Magna-IR 560 spectrophotometer fitted with an ASI DuraSamplIR ATR cell. A comparison of the spectra of the plastic specimens against the reference spectra in the Aldrich FT-IR Collection Edition II Library and Hummel Polymer and Additives library database provided the polymer identification.

Gas Chromatograph/Mass Spectrometer (GC/MS) - An Agilent 5973N GC-MSD system with an automatic injector was used in the analysis. SCAN mode was used in the identification of plasticizers in cyclohexane extracts. The mass spectra were searched against the CPSC Library and NIST98 Library database for identification. Use of the instrument in the selected ion mode (SIM) at an ion mass of 149 provided a quantitative analysis of the phthalate(s) present. The small amount of diisodecyl phthalate (DIDP), normally present in commercial DINP, was included in the reported amount of DINP.

GC conditions:

Column	30 m x 0.25 mm I.D. x 0.25 μm; HP-5MS
Injection	1 μL pulsed splitless, injector 290 °C, pulse pressure 35.0 psi, pulse time 0.5 min, purge flow 20.0 ml/min, purge time 2.0 min
Oven	50°C, 1 min, 30 °C/min to 280 °C, 15 °C/min to 325 °C
Carrier gas	Helium, 1 mL/min, constant flow

[*] This method for phthalate content was published in the Commission Briefing Package on, "Response to Petition HP 99-1: Request to Ban PVC in Toys and Other Products Intended for Children Five Years of Age and Under," August 2002, TAB I, Memorandum from Shing-Bong Chen to Warren K. Porter, "Screening of Toys for PVC and Phthalates Migration." Only the relevant portions of the "Methods" section of the memorandum are included here. In the work described in the original document, this approach for percent phthalates was applied only to DINP and DEHP, but CPSC staff believes it is a reasonable test method to extend for all 6 phthalates referred to in the CPSIA with minor modifications, as noted in the footnotes here, as part of a testing program to support General Conformity Certification.

[†] Benzyl Benzoate can be substituted as the internal standard to avoid ambiguity when testing for BBP.

Calibration – of phthalates (DINP or DEHP) for GC/MS analysis in cyclohexane[‡]

A set of solutions was prepared containing approximately 1.0, 2.5, 5.0, 7.5 and 10.0 μg/mL of DINP (Jayflex, EXXON) or DEHP (Chem Service Inc., standard grade) and 1.0 μg/ml of BBP (Chem Service Inc., standard grade). The actual ratio of DINP (or DEHP)/BBP was calculated and used in construction of calibration curves.

Calculation of % phthalate in the PVC specimen

$$\% \text{ phthalate} = [\ 100 \times C\ (\mu g/mL) \times (20\ mL \times 15\ mL)\ ] / [50\ \mu L \times mass\ (g)\ ]$$

$$\% \text{ phthalate} = [\ C\ (\mu g/mL) \times 0.6\ mL\ ] / [mass\ (g)\ ]$$

where *C* is the phthalate concentration from the calibration curve and *mass* is the mass of the PVC sample tested.

[‡] Appropriate calibration curves for each of the regulated phthalates should be created using those compounds and good laboratory practices.