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Phthalates Screening and Testing Methods

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Intertek





Our Industries



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Our organisation	Industries we operate in		What we do
Consumer Goods	Aerospace & Automotive Building Products	4	Testing
Commercial & Electrical	Chemical Consumer Goods &	9	Inspection
Commodities	Retailers Electrical & Electronic	Koterses.	Certification
Commodities	Energy Food & Agriculture	Ŵ	Auditing
Chemicals & Pharmaceuticals	IT & Telecom	T	Outsourcing
Industry & Assurance	Medical & Pharmaceutical Minerals	9	Advisory
industry & Assurance	Petroleum Toys, Games & Hardlines	只	Training
	Textile, Apparel & Footwear	Î	Quality Assurance

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- 19 laboratories worldwide accredited to Phthalates test method CPSC-CH-C1001-09.3
- " Test to other phthal ates methods (Canada, EU, etc.)
- Participate in interlaboratory studies organized by LGC Standards Proficiency Testing and Institute for Interlaboratory Studies
- Advocated for exemption of inaccessible parts recently adopted in CPSIA reform bill

Overview



- 1. Review Intertek material submission and failure rate data by material and phthalate (large sample size)
- 2. Review failure margins by phthalate (smaller sample size, more recent data)
- 3. Comments on screening in general
- 4. Comments/questions on potential screening methods

Self-Declared Materials



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Failure Rate across All Materials



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Failure Rate by Self-Declared Material



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Failure Rate by Phthalate



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Failure Margin: DEHP



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Failure Margin: DBP



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Failure Margin: BBP



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Failure Margin: DINP



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Failure Margin: DIDP



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Summary



- 1. Polymer/fiber type generally not specified by customer; some materials may be incorrectly identified
- 2. Non-compliance rate 4.2%
- 3. Most non-conformities marginal (0.1.1%).
- 4. Many non conformities in non PVC materials (glues, paints and other surface coatings, etc.)

Challenges of Screening - General



- 1. Lack of certified reference materials & uniform instrument calibration methods
- 2. Absence of screening methods (and definition of % creening method-)
- 3. Substrate complexity . polymers may contain plasticizers, fillers, colorants, flame retardants, stabilizers, lubricants which may cause interference.
- 4. Limited work on matrices other than PVC
- 5. Potential problems with small-area coatings (interference from surrounding regions?)
- 6. Possibility of Compositing
- 7. Pass/inconclusive/non-compliance boundaries as function of sample & instrument
- 8. Qualification of different instruments within a given category (e.g. sensitivity will vary with different FTIRs, sampling techniques, software packages, algorithms)
- 9. Operator experience, maintenance, interactivity, etc.

Potential Screening - FTIR



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Advantages:

- Widely available
- " Relatively inexpensive
- Possible easy sample preparation (ATR)
- % Fast (depending on sample preparation)
- Possible internal calibration using matrix absorption bands (depending on matrix/spectral complexity)
- " Potentially portable

Potential Screening - FTIR



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Challenges

- // Interference from additives
- " Relatively high detection limit
- Most sensitive sampling techniques also more time consuming (e.g. film transmission vs. ATR)
- Potential increased difficulty with complex mixtures . especially mixtures of phthalates (most phthalates are mixtures)
- Works best if library built on single machine (particularly when using difference spectra etc.)
- Qualification of different instruments / accessories / software in the field (how do algorithms differ . how well do they work with different substrates?)

Potential Screening - DART MS Questions



- DART (Direct Analysis in Real Time) invented by JEOL, sold as ion source and complete system. Few published studies on phthalates
- 2009 study¹ using single quadrupole indicates potentially high detection limits.
- What are MS recommendations for phthalates screening?
- Calibration? Source adjustments for different matrices? Interferences?

1. T. Rothenbacher and W Schwack, Rapid Comm. Mass Spectrom. 2009; 23: 2829-2835

Alternate GC-MS Techniques



" Thermal desorption, Pyrolysis, other?

- " Calibration? Interferences?
- " Potential complications related to substrates, additives?
- " Mass spec recommendations?
- " Interlaboratory studies?

Recommendations



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- // Define %creening+
- Reference materials, ideally using representative substrates and additives
- " Interlaboratory studies
- " Intertek would like to participate





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