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Determining a comprehensive methodology to screen for phthalates using portable FT-IR

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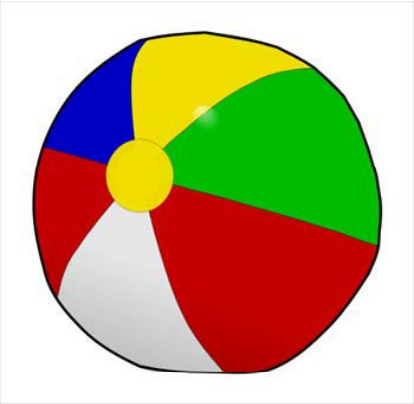
The Problem…

- Regulatory level is 0.1% (1,000 ppm)
- In PVC typically at high levels ≈ 25-30%
- Substitute plasticizers are more expensive
- Can be at <0.5% levels in some plastics, recycling/cross contamination
- There is no known field portable analytical technique that can do these levels – must lab test
  - Lab testing is time consuming, expensive and cannot possibly achieve reasonable sample rate



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The Problem…For Manufacturers

To achieve desirable rubber-like properties, concentrations of phthalates are typically between 10-35% in consumer products

"…DEHP, dominant in medical applications, is inexpensive compared with alternatives. Adipates, trimellitates, and citrates cost about 50%, 100%, and 140% more, respectively. Plasticizers can make up a third, by mass, of compounded PVC. Thus, an alternative can drive up the cost of a PVC compound from about 80 cents to about 95 cents per lb."

- ***Chemical & Engineering News*** (VOL. 83, NUM. 46, PP. 29–31)

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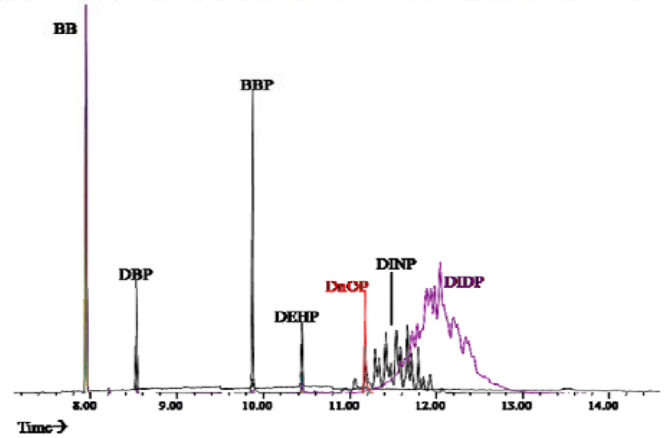
Measurement: GC-MS

- Laboratory Tool
- Need preparation
- Analysis time about one day
- Trace analysis technique
- High concentrations require dilution

Figure 1. Chromatogram overlay of all phthalates of interest and internal standard.



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FT-IR: Fourier Transform Infrared Spectroscopy

- CPSC mentions FTIR as a Pre-Screening method ~ >10%
- FTIR is a traditional laboratory technique
- Limit of detection is in the percentage range
- Useful screen for phthalates in the percent concentration range

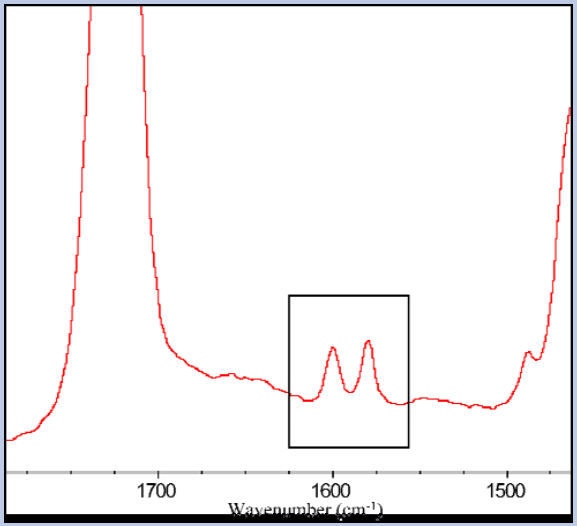

Figure 1. IR spectrum of PVC plastic containing ~30% phthalates

http://www.cpsc.gov/about/cpsia/cpsc-ch-c1001-09.3.pdf

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Background

- CPSC guidance/H.R. 2715 establish limits on phthalate content
- Acquisition of Ahura Scientific adds PFTIR Technology to TS Niton line
- First product of integrated Business Unit will address this market
  - Branded “Niton” product due to established position in this market
    - >4,000 HHXRF analyzers for lead detection in Consumer Goods
    - Standard CPSIA and Prop 65 compliance screening tool
- Existing “Ahura” product, *TruDefender*, is modified and pilot tested
  - Product name chosen - *Niton FT*

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Analysis of Market Requirements (VOC)

- Spec: Regulatory level=0.1%
  - Portable FTIR: Will detect at ~1% rang
- Spec: Ability analyze all plastics, PVC, PET, etc.
  - Portable FTIR: Will not analyze very hard materials (Diamond ATR)
- Spec: Detection/speciation of *all* Phthalates, distinguish banned from OK
  - Portable FTIR: Will detect as a group – no speciation or distinction
- Spec: Fit/Form/Function ease of use as per HHXRF standard
  - Portable FTIR: Simple operation, easy startup, long battery life
- Spec: Simple go/nogo (like HHXRF), no spectroscopist required
  - Portable FTIR: Algorithm not capable of simple pass/fail yet

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The Niton FT

- Good News
  - Phthalate screening in handheld form factor
  - Meets VOC requirements for ease performance and ease of use
  - Simple interface for non-expert use
  - Rugged equipment certified to military specifications
  - All the performance of a laboratory system in a 2.5 lb package

The Physics work very reliably

- Challenges
  - Further algorithm development needed
  - Phthalate image clearly in spectrum, but not reliably (>85%) detected by algorithm
    - Algorithm not specifically written for phthalates

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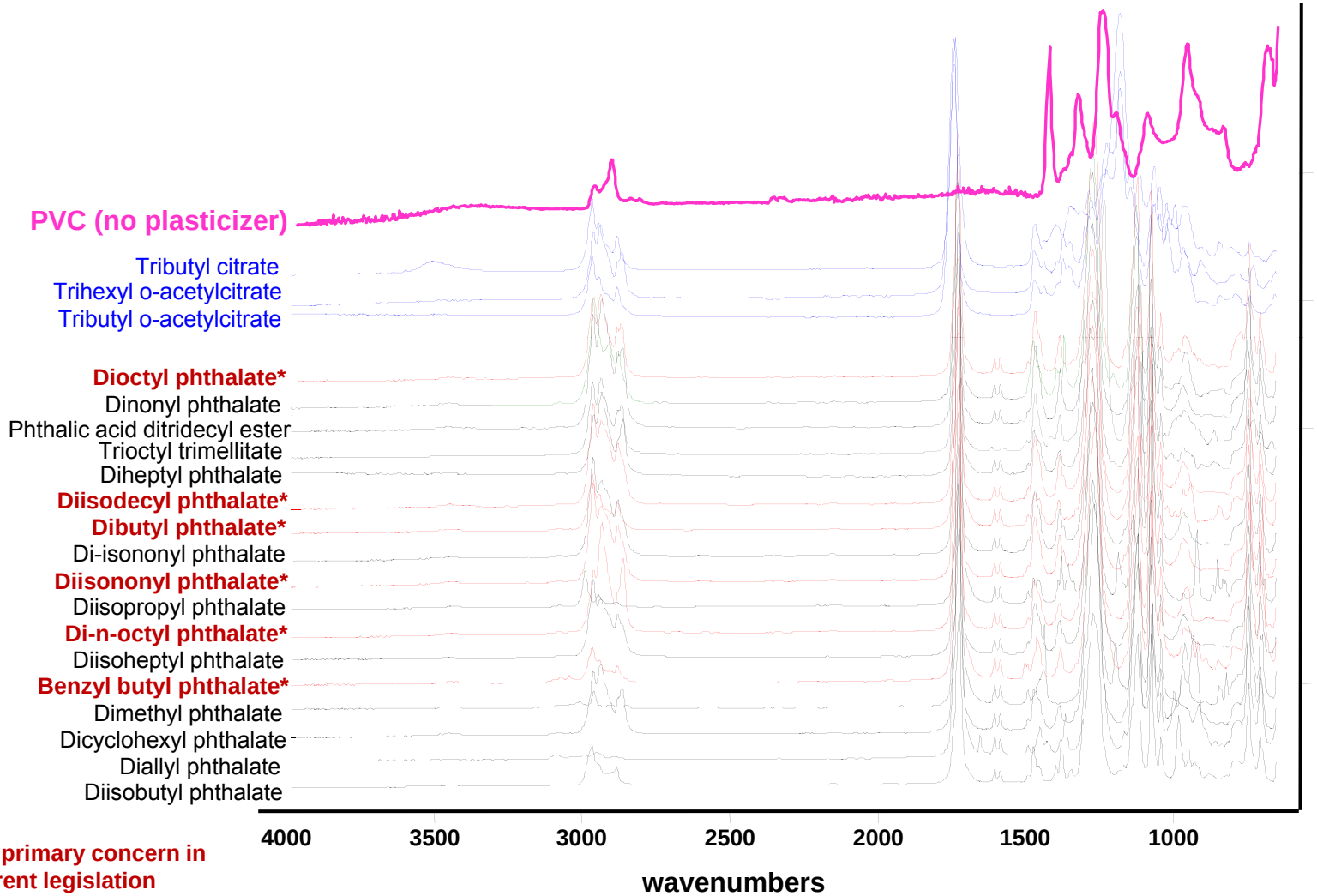
Each phthalate has a distinct FTIR spectrum



*** of primary concern in current legislation**

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Summary

- Need is still developing
  - H.R. 2715 in August of 2011
  - 2011 first year phthalate surpasses lead on 60 day notice list
- Portable solution is the only chance for statistically meaningful testing
- Product has existed in present physical form for 3 years
  - Several thousand delivered
- Pilot showed:
  - Need more algorithm work
  - Need better pass/fail functionality
  - Ability to run most common materials, some limits on very hard plastics
  - Solid F/F/F
  - Solid physics

The Physics work very reliably

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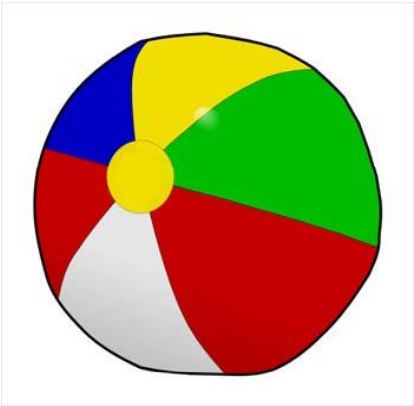
Thank you



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