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Exposure to chemicals from toys: case of phthalates in soft PVC toys.

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Background

- ★ **Phthalates have typically been used as softeners in soft PVC toys and childcare articles.**
- ★ **There have recently been renewed health concerns regarding these substances**
  - ➢ **Need of estimating the in-vivo potential exposure of infants to a variety of phthalates via mouthing of such toys**
  - ➢ **Decision either to ban or set limit of acceptable release (migration)**
  - ➢ **Developing the means to test these toys or childcare articles using mechanical/analytical means for quality or enforcement purposes.**

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Legislative context

- **Directive 76/769/EEC**
  - approximation of the laws, regulations and administrative provis ions of the Member States relating to restrictions on the marketing and use of certain dangerous substances and preparations
- **Directive 88/378/EEC**
  - on the approximation of the laws of the Member States concerning the safety of toys
- **Directive 92/59/EEC General Products Safety (Dir. 2001/95/EC)**
- **Decision 1999/815/EC**
  - adopting measures prohibiting the placing on the market of toys and childcare articles intended to be placed in the mouth by children under three years of age made of soft PVC containing one or more of the substances di-iso-nonyl phthalate (DINP), di(2-ethylhexyl) phthalate (DEHP), dibutyl phthalate (DBP), di-iso-decyl phthalate (DIDP), di-n-octyl phthalate (DNOP), and butylbenzyl phthalate (BBP)

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Area of toys/childcare articles

- **Large variety of materials**
  - Wood, polymers, coated metals, textiles, rubber etc
- **Large number of substances**
- **Little knowledge of composition**
- **Little knowledge of manufacturing specifics**
  - E.g. PVC rotocast, injection molding, sheet
- **Large number of toys "categories"**
  - Bath toys, activity toys, books, action figures & dolls, vehicles, games/puzzles, infant, plush, ride-ons etc.

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Scientific aspects (1)

★ **Health concerns for substance(s)**
- Toxicology of the substance (In-vivo, in vitro tests)
- Tolerable daily intake (TDI)
- Role of the European Chemical Bureau

★ **Extent of release of substance(s) from mouthing on toys**
- In-vivo studies to obtain release rates
- Studies only possible on adult volunteers
- Definition of mouthing: sucking only or also biting??

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Scientific aspects (2)

★ **Consumer behavior of exposure**
- How much time do babies spend on sucking (biting?) on toys
- as a function of age group (hence body weight and associated risk)

★ **=> Exposure of the consumer (in µg/day or risk of absorption in µg/kg body weight)**
- compared to the tolerable daily intake (TDI)

★ **Implication of legislation type on methodology**
- Ban => extraction (LOQ)
- Migration limit => migration + extraction

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Migration

- If in-vivo study can lead to a release value in standard conditions that can be used as a reference
- If toxicology data shows mild concern
- Then a limit of release (migration) rather than a ban (extraction) can be considered
  - Testing of toys left to enforcement laboratories,
  - Methods for migration must be agreed or developed
  - Tests must show an acceptable performance among laboratories.

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Uncertainties: in-vivo studies

- Base for target rate of release
- Experiments: TNO (DCS, NL) and CPSC (USA)
- Adult vs. children
  - pH, saliva composition, jaw strength
- Mouthing: sucking vs. also biting
  - Spit and extract:
    - Chips of materials
    - Dissolved by extraction solvent
    - Dispersion of results
    - Spit-extract vs. swallowed-excreted
  - Averages and extremes
- Absorption through buccal membranes

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Uncertainties: consumer behaviour

- ★ Infants' behaviour
- ★ Experiment U. Wagenigen (DCS)
- ★ Most easily modified by external presence or unfamiliar location
- ★ Compromise
  - ➢ Systematic measurements
  - ➢ In-situ observations.

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Uncertainties: migration

- ★ Field of food contact materials
  - ➢ Migration by diffusion processes
  - ➢ t/T exposure under worst foreseeable conditions
- ★ Toys (case of phthalates from PVC toys)
  - ➢ Solubility of substance in saliva very low
  - ➢ Mouthing => in vivo release > migration by diffusion
  - ➢ Need dynamic migration methods to simulate in-vivo
- ★ Mechanical simulation of in-vivo action of mouthing
  - ➢ To target levels
  - ➢ Reproducibility of methodology for acceptance by MS
  - ➢ Sucking or also biting?

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Implication of migration limits

- Impediment to developing legislation restricting by migration limits in the end rested on a method that would ensure test results on toys could be trusted with a safety margin adequate to ensure acceptable exposure uncertainty and consequently consumer safety.

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Process of method validation

- JRC co-ordination
- Validation of methodologies to measure the release of phthalates from toys
- Tested on DINP because most commonly used in toys/childcare
- Methods applied to
  - Variety of toys
  - different manufacturing processes
  - various levels of DINP

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Methods tested

★ **Horizontal shaking (UK)**
- ➢ Variable stroke, amplitude
- ➢ Heated
- ➢ Stringent conditions
- ➢ Mild conditions

★ **Head over heels (NL)**
- ➢ Rotation over axis
- ➢ Mild conditions

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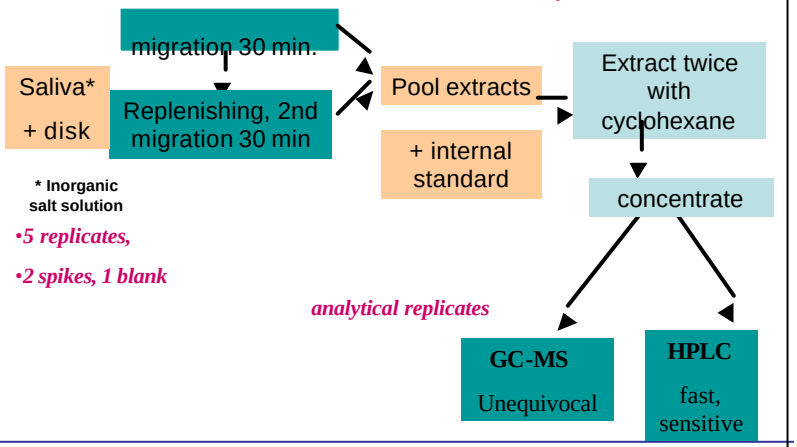
Harmonised extraction/analysis

~~Harmonisation based on research done by JRC~~



* Inorganic salt solution

•5 replicates,

•2 spikes, 1 blank

analytical replicates

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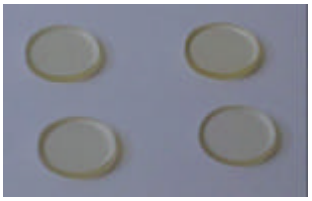
Reference materials

- PVC known composition
- Dutch Consensus Group
- 38% of DINP
- Homogeneity of materials



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Choice of toy materials

- Not necessarily made for mouthing
- For <3yrs and >3yrs of age
- Range 20-50% of phthalate content
- Allow >1 phthalates (applicability)
- Cover manufacturing processes
- Toy surface big enough to punch 5 specimens
- At least 5 toys
- Some high “foreseen release”of DINP

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Toys and sample preparation

Sample preparation: punching

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Results (HoH):

★ Spikes (recoveries)

	5µg/ml		25µg/ml	
	GC-MS	HPLC	GC-MS	HPLC
MEAN	**92.95**	**92.57**	**92.78**	**94.65**
n	13	10	12	10
RSDr	**3**	**3**	**2**	**2**
RSDR	**16**	**14**	**16**	**12**

Mean: average of valid results
n: number of laboratories
RSD_r: repeatability relative standard deviation
RSD_R: reproducibility standard deviation

★ Results

	disks		Worm		Nikki		Duck		Betsy		Tiny	
	GC	LC	GC	LC	GC	LC	GC	LC	GC	LC	GC	LC
MEAN	**3.72**	**4.06**	**6.15**	**4.89**	**4.32**	**3.80**	**5.17**	**6.07**	**1.74**	**2.36**	**3.20**	**3.46**
n	14	12	14	12	13	12	11	12	12	12	12	12
RSDr	**6**	**6**	**6**	**5**	**6**	**10**	**6**	**4**	**8**	**7**	**8**	**6**
RSDR	**28**	**33**	**43**	**35**	**60**	**65**	**58**	**48**	**41**	**53**	**41**	**50**

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Results

- **HOH: lower RSDR (best reproducibility)**
  - RSDR ~30% on reference disks
  - Toys: 40 to 65% for toys tested in this study.
- **Horizontal shaking:**
  - Problem of reproducibility and results (<8)
  - Mild: RSDR ~90% for disks, 62-110% for toys
  - Stringent: RSDR ~140% for disks, 65-115% for toys

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Conclusions

- **HOH: validated**
  - RSDr 4-9%, RSDR 30% (disks), 35-65% (toys).
- **Migration of food packaging:**
  - Diffusion (t/T exposure)
  - CEN methods: RSDr 21%, RSDR 38%,
  - CEN solely using standard reference materials => RSDR of 30% on disks acceptable.
- **Dispersion also depends on toy nature**

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