Summary of Regulatory and Standard Method Activity Relating to Testing Materials Emissions to Indoor Air

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Construction Products Directive

- European Council Directive 89/106/EEC
- Essential Requirement No.3; Hygiene, Health and the Environment
- a healthy indoor environment can be achieved by the controlling of sources and by eliminating or limiting the release of pollutants into the air

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CPD; Guidance Paper H on Harmonised Approach relating to Dangerous Substances, DGIII, 2001

- The requirement is expressed either as emission or migration of dangerous substances.
- Wherever possible it is the release of substances (not the content itself) that should be controlled
- Specification writers should identify all dangerous substances in mandate and/or the Commission database (Annex 2)
- Specification writers should define thresholds (or possibly classes)
- As far as possible use horizontal test methods to determine emission of dangerous substances
- if necessary, product standards can complement test method by provisions on sampling and preparation of specimens
- Complete harmonisation not always possible in short term (apply state of the art)

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Main ‘horizontal’ standards for VOCs

- (Formerly…) ENV 13419 (CEN TC 264, WG 7)
  - Part 1 – emission test chamber
  - Part 2 – emission test cell
  - Part 3 – selection and preparation of samples for testing
- ISO 16000 (ISO TC 146, SC6)
  - Part 6 – Tenax tube, TD-GC-MS(/FID) analysis – full ISO std
  - Parts 9 to 11 – almost identical to what was ENV 13419 parts 1 to 3 respectively. Now fully approved as EN/ISO
- *Other parts of ISO 16000 (now up to #24) include:*
  - *Sampling strategy (VOCs & formaldehyde)*
  - *Determination of formaldehyde/carbonyls (active & passive)*
  - *Characterisation of ventilation*
  - *Measuring particulates including asbestos and moulds*
  - *Sampling and measuring semi-volatiles such as (PCDDs/PAH/PCDFs)*
  - *Testing sorptive materials (formaldehyde and VOCs)*
  - *Sampling NO_2*

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Process of VOC Emissions Testing

- Tests for VOC content are justifiably considered unsuitable / not-representative of VOC emissions
- The reference test methodology, developed over the last ~15 years involves:
  1. Selection of representative product samples from manufacturers or store and transport of sealed samples back to lab
  2. Preparation of test specimen from sample
  3. Introduction of test specimen to simulated indoor environment (test chamber or cell) swept with pure, humidified air. Orientation? Time of introduction equates to t_0. Chamber maintained at fixed temp throughout – typically 23C
  4. Sample of air-borne vapours collected at various times after t_0 – typically 3 days and 28 days. Carbonyls collected on DNPH cartridges for subsequent HPLC analysis. Other VOCs collected on Tenax tubes for subsequent analysis by Thermal Desorption with GCMS.
  5. TD-GCMS system calibrated with authentic standards of target compounds. Allowing levels of these compounds to be identified and measured in the collected vapour samples. Other (non-target) compounds can often be indentified and their concentration estimated
  6. Suitable for compounds ranging in volatility from n-hexane to n-hexadecane and sometimes extrapolated outside this range

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Test chamber with a sample of flooring in a sealed box

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Another example of chamber sampling

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Emission Test Chambers/Cells

Simulation of a small room of 7 m² and 2.5 m height

- Size: typically 100 - 1 000 litres
- Alternative versions (different air velocity):
  - FLEC cell: very small, 35 ml
  - Microchamber: 3 or 40 ml
- Temperature: 23 ℃ (but Japan: 28 ℃, Korea: 25 ℃)
- Relative Humidity (50 %)
- Air exchange / Loading
  = Area Specific Flow Rate
  - Take data from the exposure scenario

FLEC Cell – Suitable for planar surfaces (EN/ISO 16000-10)

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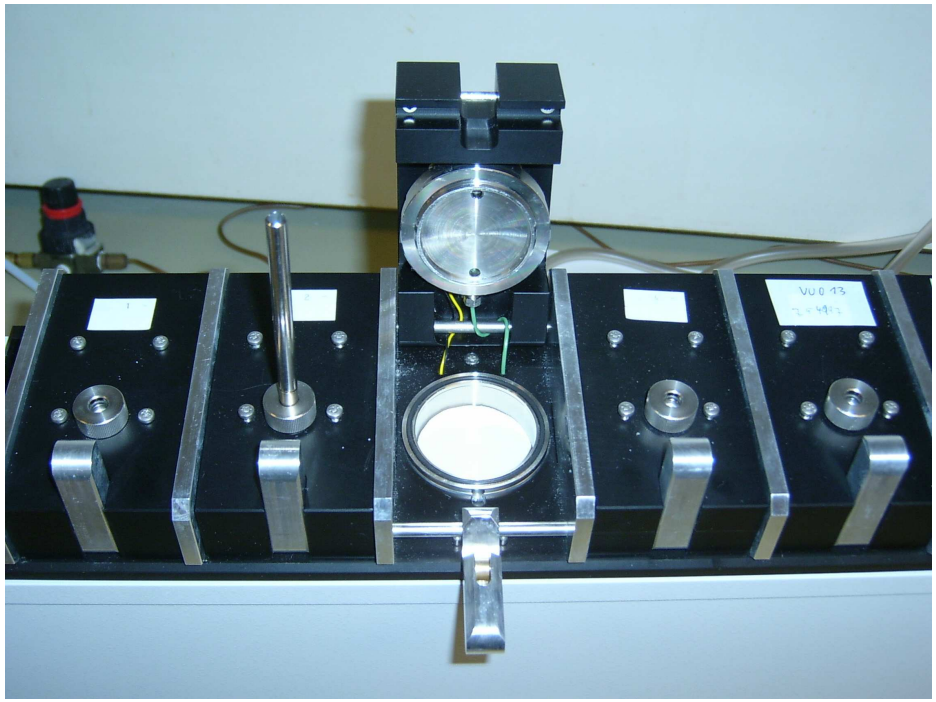
Microchamber

Volume: 3 ml or 40 ml

(depending on operation mode)



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Air sampling

2 Tenax TA tubes in series

2 tube sets in parallel

= 4 tubes per analysis

Photo: Eurofins Environment A/S

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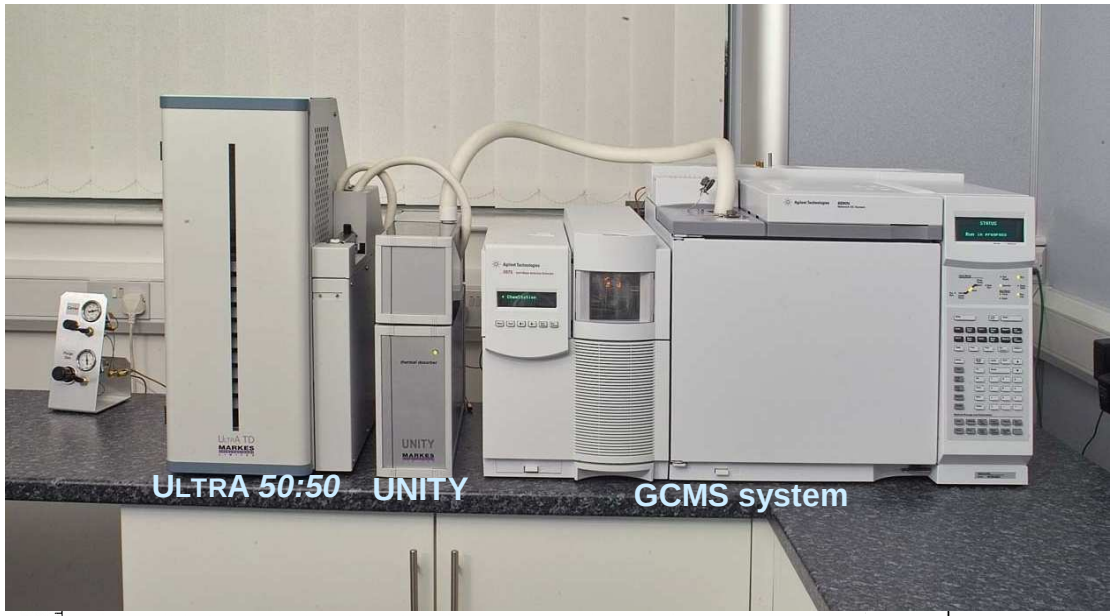
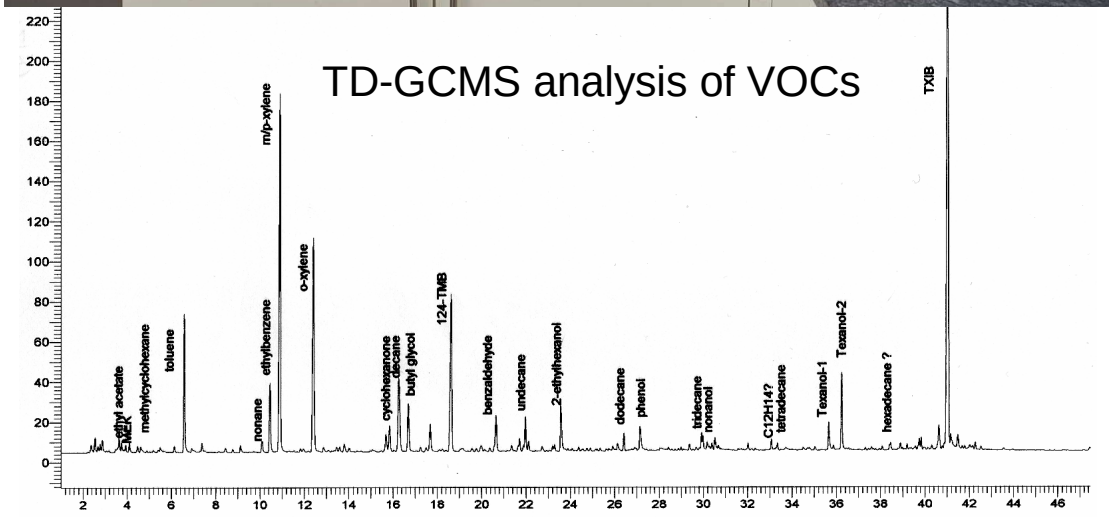
Analyses

TD-GCMS: VOC, TVOC, VVOC, SVOC

HPLC:
Aldehyde,Isocyanate





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ISO 16000-6; VOC measurement

- Calibration is by liquid or standard atmosphere spiking of sorbent tubes
- Air sampling flow of 50 to 200 ml/min (indoor and chamber air) onto Tenax TA tube
- Compounds identified by GCMS, quantification by FID or MS. Unidentified compounds quantified using detector response to toluene
- Quantify as many VOC as possible, particularly 10 highest and those above 2 $\mu g/m^3$ by FID or MS
- Informative annex listing 171 compounds often detected in indoor or chamber air
- Total volatile organic compounds; sum of VOCs sampled on Tenax TA and eluting between and including n-C_6 and n-C_{16}, detected with an FID (TVOC FID) or MS (TVOCMS) and quantified as toluene equivalents.
- VVOC and SVOC observed outside the TVOC range should be identified if possible and summed (toluene equivalent) concentrations reported
- A ‘note’ refers to ISO16017-1 for alternative methods for VOCs outside TVOC range.
- Includes some validation (overall uncertainty) for measuring some VOCs in indoor air (not in relation to chamber testing)

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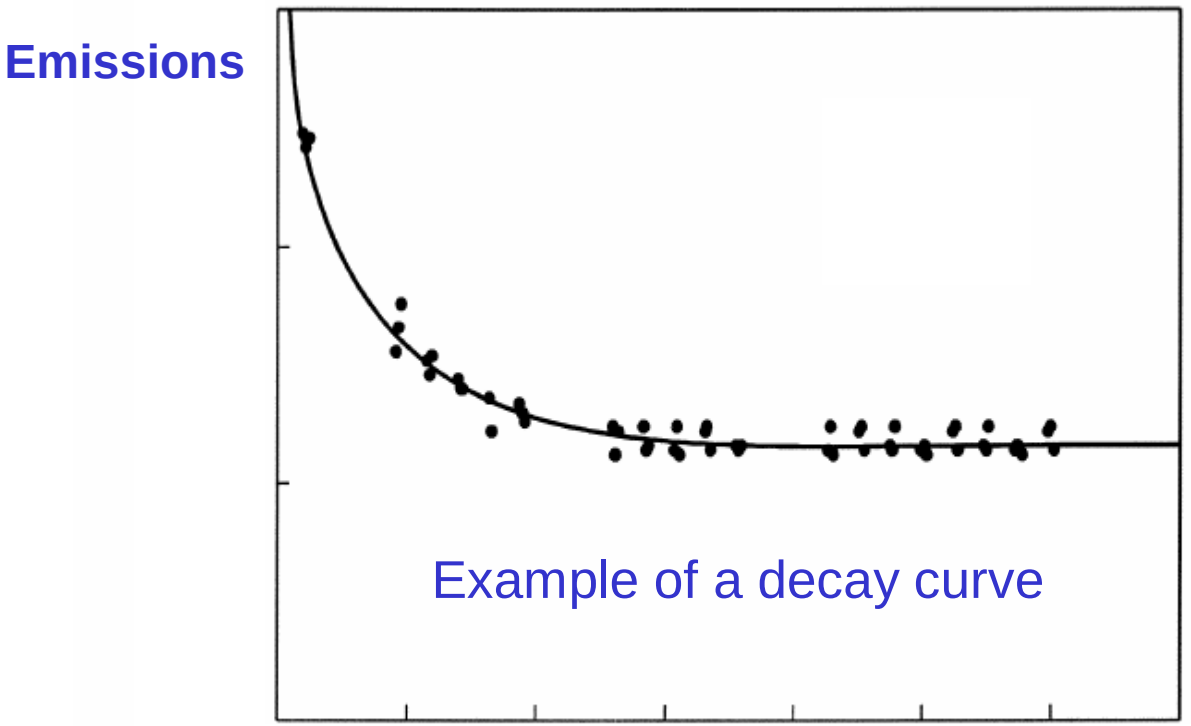
VOC Emission Decay Over Time



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Mandate M/366 (03/2005) under Essential Requirement 3 of the 1989 Construction Products Directive (CPD)

- Prepared by EC/DG Enterprise and agreed by Standing Committee on Construction November 2004
- The objectives of M/366 are to validate existing horizontal methods for testing emissions of dangerous substances under the CPD (for indoor air this means EN/ISO 16000) and thus facilitate them being cited in the CPD regulations – Effectively this will make them a mandatory part of the CE marking for relevant products. (M/366 also allows new standards to be developed if the existing horizontal methods are not deemed suitable.)
- To develop/validate complementary quick test methods for emissions screening / initial-testing / content testing
- To work on the above in the context of a list of 'regulated dangerous substances' provided by the commission. Currently, the list of dangerous substances for emissions to indoor air comprises 'VVOCs', 'VOCs', SVOCs', some specific inorganic gases (e.g. ammonia), metals and asbestos fibres
- Target date for completion is 2008

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CEN TC 351 – Emission of Regulated Dangerous Substances from Construction Products

- CEN TC 351 was set up to implement EC mandate M/366 under NEN (NL) convenorship.
- WG 1 deals with leachate to soil/water and WG 2 deals with emissions to indoor air. Chair of WG 2: JF Vicard, AFNOR (France).
- CEN TC 351 WG 2 overlaps to some extent with CEN TC 264 (ambient air) WG 26. [NB CEN TC 264 is the committee originally responsible for EN/ISO 16000 parts 9-11.] The chair of CEN TC 264 WG 26 is also JF Vicard
- Process:
  - List relevant available standard methods
  - Decide which most appropriate for fulfilling requirement of M/366/CPD ER3 (Presumably EN ISO 16000 parts -6, -9, -10, -11)
  - Validate same with appropriate round robin testing

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Other activity in ISO TC 146 and CEN TC 264

- ISO TC 146 SC6 proposed the following additional work items at its meeting in Philadelphia (10/06):
  - Emissions of SVOCs using micro-chambers
  - Reference methods for car interior emissions testing:
    - Whole car
    - Chamber
    - Cell
- CEN TC 264 WG 26 recently extrapolated the scope of emissions testing from construction products to:
  - ‘and furnishings’
  - ‘and other equipment’

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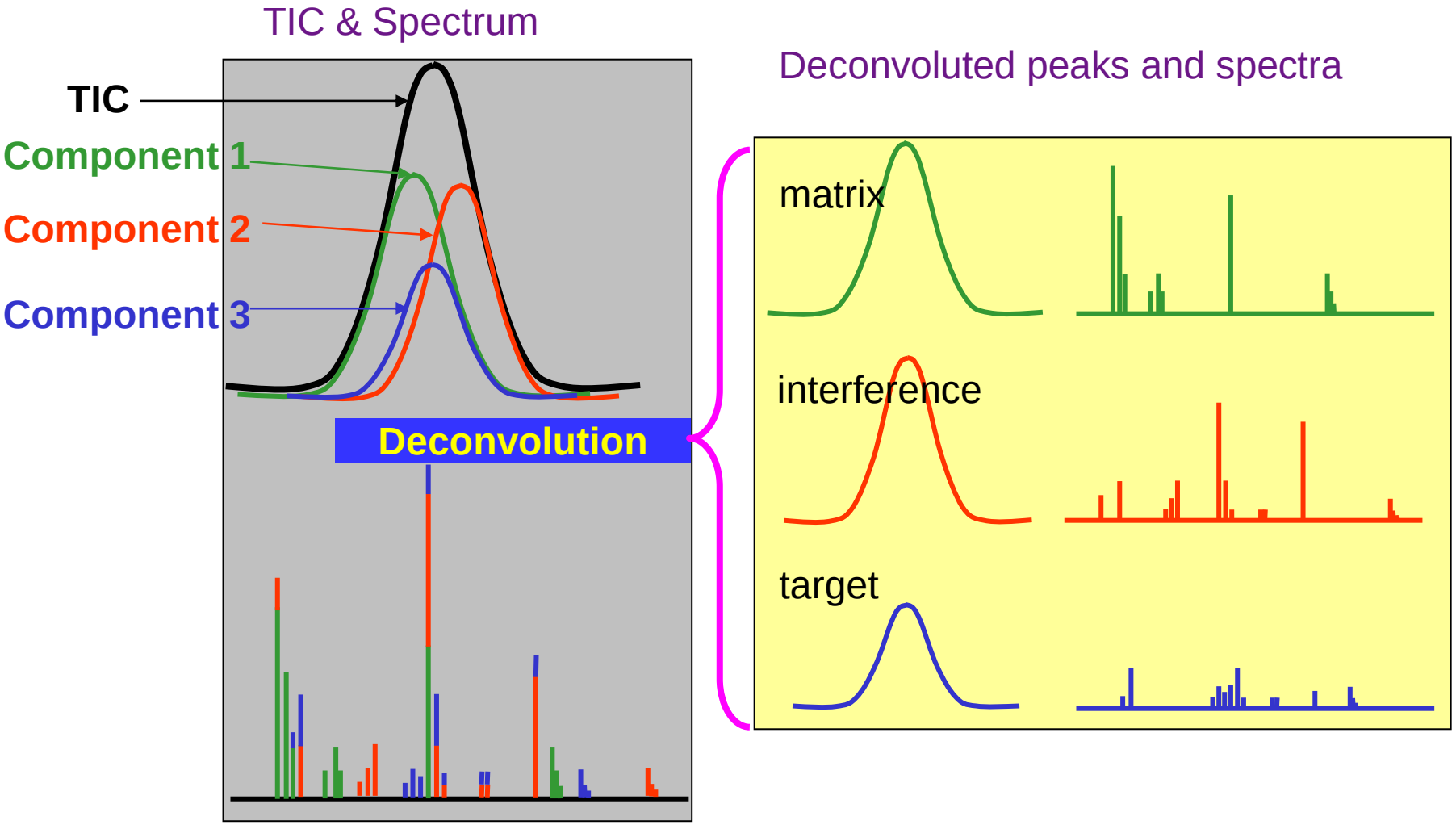
Other related activity in individual EC Member States – Germany

- Germany now has a national regulation requiring VOC emissions testing from fire-resistant floor coverings (inc imports) using a protocol known as the AgBB scheme. This can only be carried out by DIBt-accredited labs.
- The German AgBB protocol is a construction product certification procedure based on EN/ISO 16000 parts -6 and -9, but also including SVOCs
- It specifies test times, components of interest - LCI's (compounds with an workplace limit level) and carcinogens - and pass/fail criteria in terms of individual compounds plus total-VOC and total-SVOC
- Concerns:
  - Appears to require calibration with individual compounds of interest (all 400 of them!!).
  - Users currently directed to internet for updated list of carcinogens
  - Specifies total carcinogen limit values lower than individual compound detection limits
  - Some of the listed compounds are not trapped on Tenax
  - Will require the ultimate in TD-GCMS data processing
- DIBt plan to extend to other product groups

AMDIS (NIST) deconvolution separates individual components by their spectra – DRS automates this process



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AMDIS allows mass spectral deconvolution of co-eluting peaks improving detection of trace target compounds

Other related activity in individual EC Member States - France

- The French Agency AFSSET (Avis de l'Agence Francaise de Securite Sanitaire de l'Environnement et du Travail) has just published a similar material emissions test protocol to the German AgBB scheme.
- It applies to all solid materials such as interior walls, ceiling tiles, floor coverings, wall coverings, wall insulation materials, etc
- It too is based on ISO 16000 parts -6 and -9
- Differences between AFSSET and the AgBB scheme include:
  - AFSSET does not include SVOCs
  - Most French 'Lowest Concs of Interest '(LCIs) are lower than German equivalents (i.e. some products could be accepted under AgBB but rejected under AFSSET)
  - Applies to mutagens and reprotoxins as well as carcinogens and has the same issue as the AgBB scheme with respect to pass/fail criteria i.e. sum of all CRMs currently being specified lower than detection limit for each individual compound
- There is no French national regulation requiring this test to be carried out

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Other related activity in the ‘product’ TCs

- **CEN TC 134 (flooring)** responded quickly to the AgBB scheme by developing their own standard (prEN 15052) for emissions testing of textile, resilient and laminate flooring. Championed by ECRA, based in Brussels, this draft std has now gone for final CEN enquiry. It follows a similar approach to the AgBB scheme but specifies a simpler calibration regime and that no carcinogen should be ‘above the determination limit level’.
- **ISO TC 219 (flooring)** – ISO TC 219 are now collaborating with those working on prEN 15052 and have produced an ISO Working Draft Document for measuring VOC emissions from flooring. This is out for preliminary ISO enquiry now
- **Adhesives** already have their own emissions testing standard EN 13999. It too is based on principles similar to EN/ISO 16000 parts -6 & -9
- **OTHER – for example toys** – EN 71-11 Annex A2 - testing vapour phase emissions from toys materials using thermal desorption – **and cars** – VDA 278 direct thermal desorption and VDA 276 heated chamber

And outside the EU……??

- Arnold has endorsed the **California CHPS program** and associated 01350 protocol for materials emissions testing. This has a different test scheme than any other protocol, but works along the same lines as the AgBB and AFSSET protocols, with pass/fail criteria, extensive compound lists and referencing of users to the internet.
  - Also requires accredited labs and ANNUAL certification testing
  - Being adopted by other US States
  - Protocol was proposed as ASTM work item – status unknown
  - Competitor to Greenguard / Greenguard+
  - Industries potentially affected: - flooring, paints, furniture, levelling compounds, wall coverings, sealants, adhesives, wood and wood-composite products, etc, etc
- **Japan, China and Korea** has limits for formaldehyde and have or are introducing emission limits for 12-13 compounds from car cabin trim components

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Meanwhile back with the Commission

- **REACH !!!!!!!!**
- Buried within the 1200 pages of the October 2003 version of REACH lies the following paragraph (Section 2.2, Article 6):
  - '***Manufacturers or importers shall also notify the Agency of certain specified information if substances contained in articles meet the criteria for classification as dangerous, are known to be released during normal and reasonably foreseeable conditions of use even though this is not an intended function of the article, in quantities that may adversely effect human health or the environment…'***
- Hence toys and cars may not be the only non-construction related product group to try to produce their own method for testing material emissions to indoor air

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Example ASTM standards in this field

- **D 5116** Standard guide for small-scale environmental chamber determinations of organic emissions from indoor material/products
- **D 5172** Test method for determination of formaldehyde and other carbonyl compounds in air (active sampler methodology)
- **D 6196** Standard practice for selection of sorbents, sampling and thermal desorption analysis procedures for VOCs in air (and material emissions chambers)
- **D 6670** Standard practice for full-scale chamber determination of VOCs from indoor materials / products
- **D 7143** Standard practice for emission cells for the determination of VOCs from indoor materials / products
- **D 6330** Standard practice for the determination of VOCs (excluding formaldehyde) emissions from wood-based panels using small environmental chambers under defined test conditions
- **D 6803** Standard practice for testing and sampling of VOCs (including carbonyl compounds) emitted from paint using small environmental chambers
- **D 6177** Standard practice for determining emissions profiles of VOCs emitted from bedding sets
- **WK 3464** Standard test method for determination of VOCs in carpet using a specific sorbent tube and thermal desorption / gas chromatography

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Simpler options for emissions screening / initial testing

- Results should correlate with emission test chamber or cell
- More simple tests:
  - Contents, e.g. by extraction
  - Evaporation of formaldehyde (VDA 275 and more),
    Fogging (ISO 6452),
    Headspace (VDA 277), in-can (ISO 17895)
  - Thermal desorption / extraction (VDA 278)
  - Microchamber (ISO 16000)
- Analyses: Same as for emission test chamber

Which of these may substitute chamber testing under which presumptions?