The long and bumpy rood to harmonised VOC measurement and evaluation of emissions

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IEH Institute of Environment and Health

Are we nearly there?

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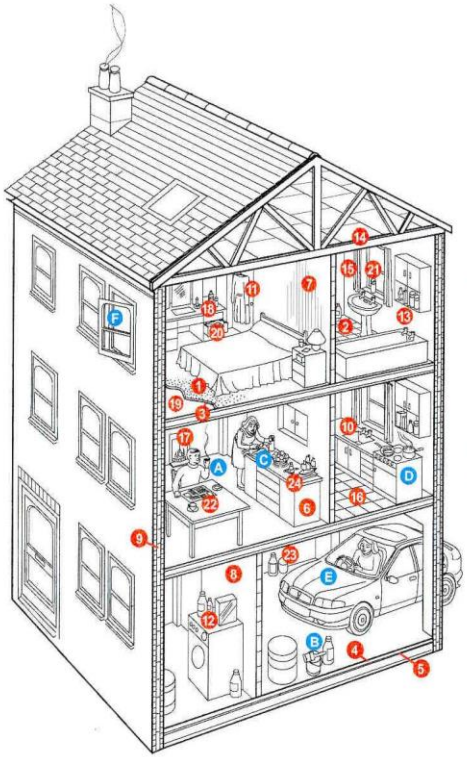
Indoor pollutant concentration

Concentration of pollutant indoors depends on level in outdoor air, release into indoor air, removal by ventilation, sorption to surfaces and reactions



ACTIVITIES

- A Cigarette smoking
- B Painting of windows, doors etc.
- C Furniture polishing
- D Cooking
- E Car exhaust/petrol vapour
- F Ventilation (outdoor air pollutants)

PRODUCTS

1. Carpet
2. Vinyl flooring
3. Carpet underlay
4. Self-levelling screed
5. Liquid applied damp proof membrane
6. Particleboard furniture
7. Wallpaper
8. Walls painted with emulsion
9. Cavity wall insulation
10. Sealant around worktop in kitchen/bathroom
11. Dry-cleaned clothes
12. Store of cleaning materials
13. Toiletries in bathroom
14. Timber in joists
15. Curtains
16. Vinyl adhesive
17. Open fire
18. Perfume
19. Chipboard flooring
20. Moth repellent
21. Air freshemer
22. Printing ink
23. Pesticides
24. Glues for hobby

Indoor Product Sources

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- Volatile organic compounds (VVOC, VOC, SVOC). e.g. formaldehyde, phthalates
- Particles (e.g. PM10, fibres; contain metals and organics),
- Gases including radon
- Emitted to air and dust
- Airborne and sorbed to surfaces; inhaled and ingested
- Source control combined with appropriate ventilation and outdoor AQ achieves good IAQ

Traditional materials

- Standen - late Victorian family home and garden hidden at the end of a quiet Sussex lane.
- Morris & Co. interiors, set in a beautiful hillside garden.

Drivers for control of emissions

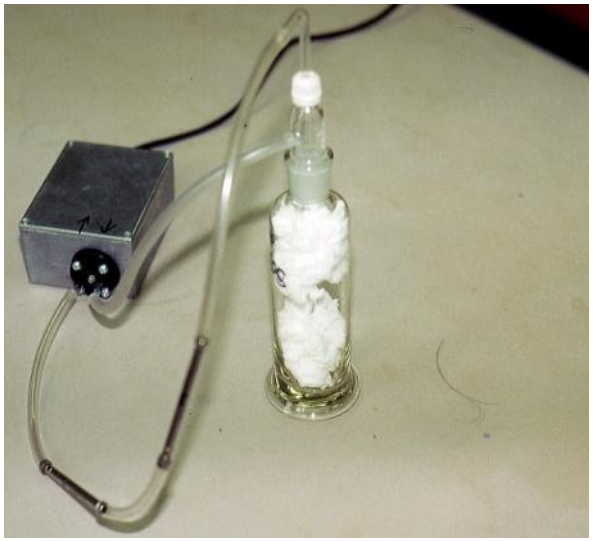
Growing awareness of;

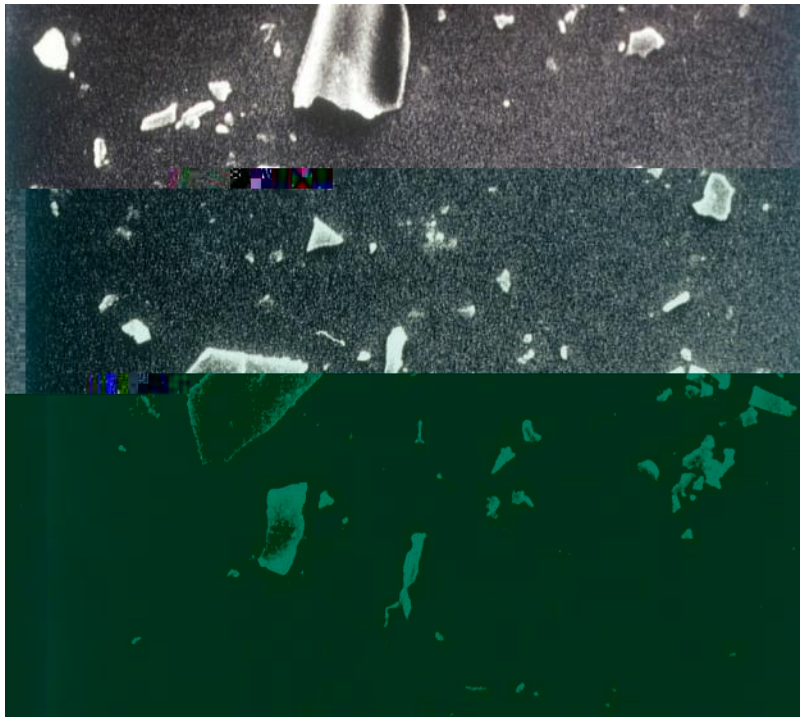
- the benefits for IAQ of reducing source emissions particularly when ventilation is reduced to save energy costs
- the important role of the indoor environment in determining the total exposure of people to a wide range of pollutants and associated adverse health and performance impacts

IAQ problems e.g.

- Urea formaldehyde foam insulation
- Damp proofing materials
- High solvent containing products
- Wood based products with ‘high’ rates of formaldehyde release

UFFI – 1980s





Low emitting products

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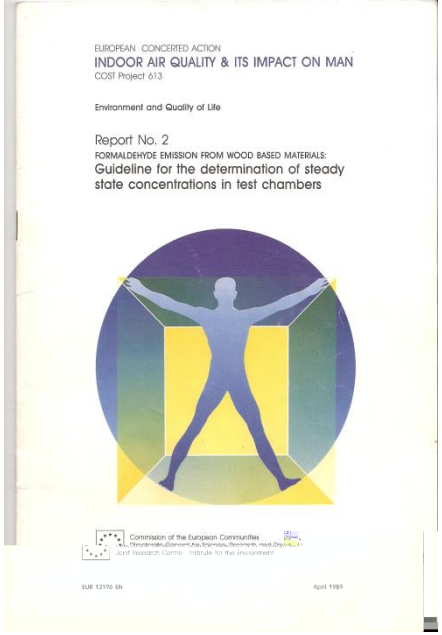
My Definition

A product that does not cause exposure to an agent that adversely affects the health and well being of building occupants.

Issues

- Inadequate knowledge of exposure and health for many agents
- What level of risk
- Protection appropriate for children, elderly, sick and healthy adults.
- Different standards appropriate to building type?
- Is it the product or its conditions of use that are the problem
- Accounting for presence of multiple products
- Product must still perform
- Marketing tool or health benefit

Testing formaldehyde emission from wood based products - ECA 1989 (report no. 2)


EUROPEAN CONCERTED ACTION
INDOOR AIR QUALITY & ITS IMPACT ON MAN
COST Project 613

Environment and Quality of Life

Report No. 2
FORMALDEHYDE EMISSION FROM WOOD BASED MATERIALS:
Guideline for the determination of steady state concentrations in test chambers

Commission of the European Communities
Joint Research Centre - Institute for the Environment

EUR 12196 EN

April 1989

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- Conditions 1ach, 23˚C, 45%RH,
- Loading ratio of 1 m^2 m^{-3}
- Determine steady state concentration
- Minimum test period of 10 days and maximum of 28 days
- Evolved into standard EN 717-1 for wood based products
- No VOC measurements

Formaldehyde concentration in 1m^3 chamber

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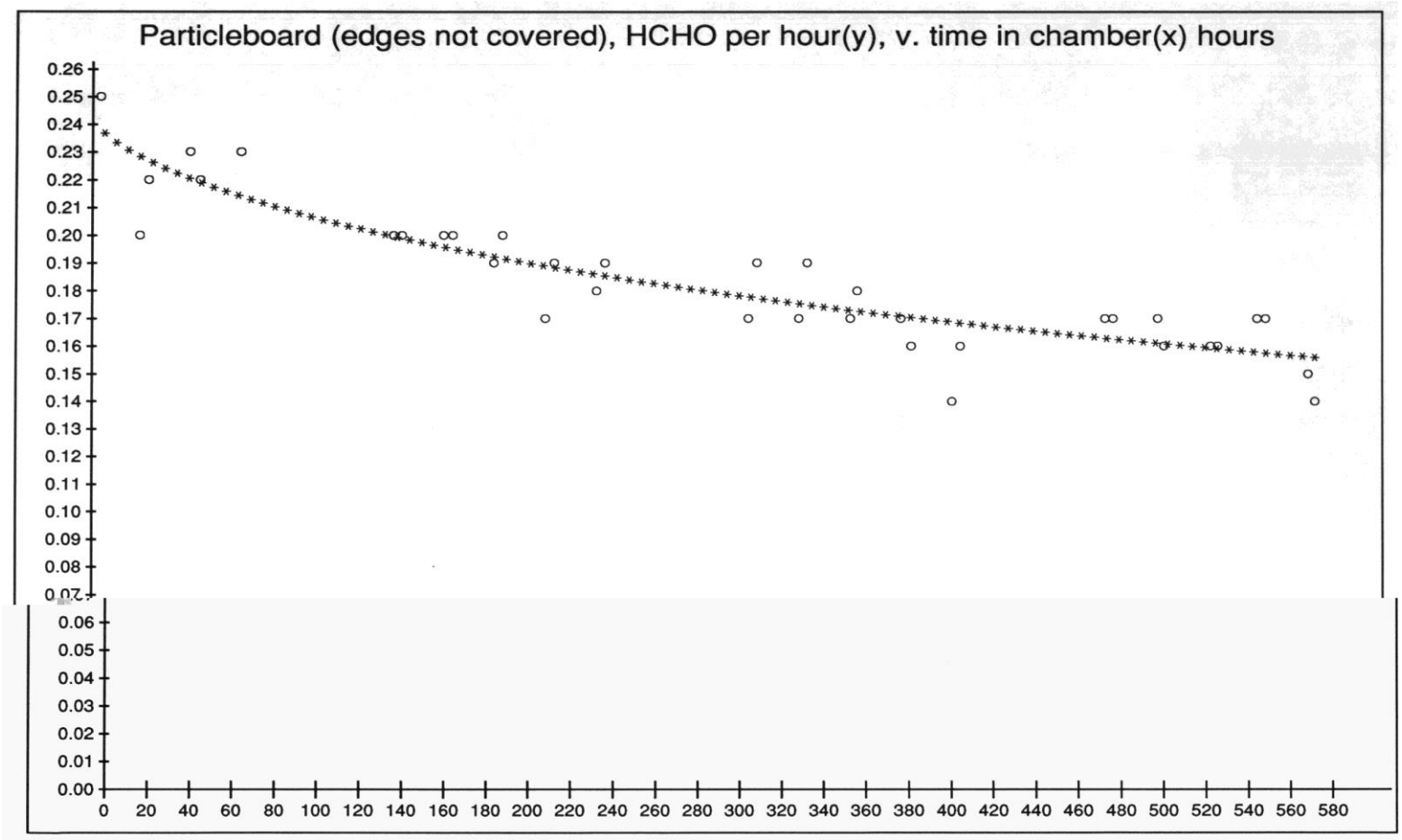

Testing of VOC emissions from materials – ECA 1991 (report no. 8)

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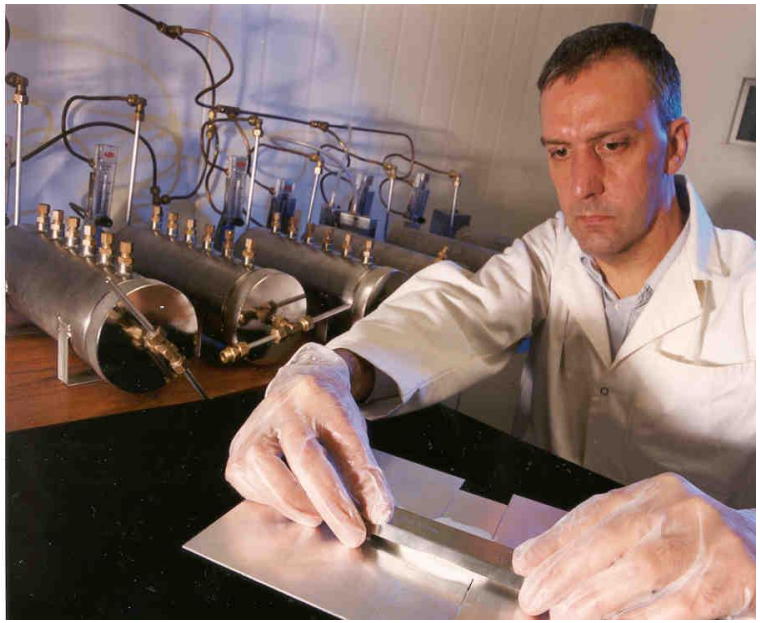
- Expert EU group recommendations
- Environmental test chamber characteristics
- Air sampling and analysis
- Recommended test conditions;
- 0.5 or 1 ach, 23˚C, 45%RH
- Realistic product loadings e.g. flooring 0.41 $m^2.m^{-3}$
- Reporting of emission rate mg m^{-2} h^{-1}
- Screening tests (microchambers)



Development of standards

- ECA on IAQ round robin studies of chamber testing (1993-1999) and proposed a system of evaluating emissions using flooring as an example (ECA report 18 in 1997)
- CEN TC 264 WG7 initiated work to develop standards on emissions of VOCs from building products in 1993
- In 1999 ENV 13419 parts 1-3 published on emission testing using chambers and cells
- Subsequently revised and published as EN ISO standards in 2006
- ISO TC 146 SC6 WG3 developed analytical method standard for VOCs; ISO 16000-6: 2004

ECA report 21; 1999

Third interlaboratory comparison of emission Testing using pre-standards (EN ISO).

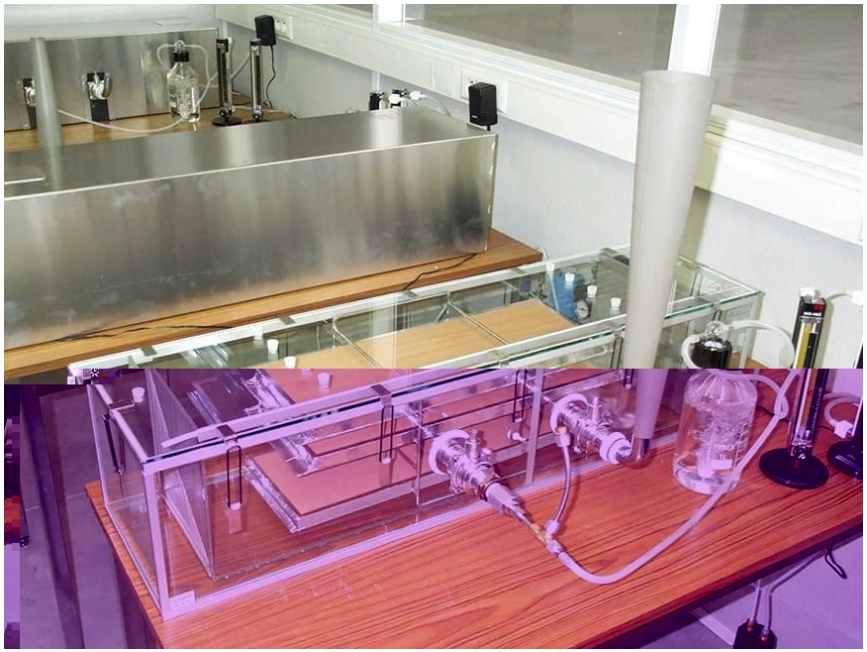
- The between laboratory factor makes by for the largest contribution to overall variance – the analytical quality of labs should be verified
- In addition to analytical errors, non-homogenity of materials contribute to interlab variance; even for same batches of carpet and PVC
- Need improved methods of preparing standardised paint films
- 3rd important cause of variance is VOC sorption to chamber walls

European and international test standards

- EN ISO 16000-9; Emission test chamber
- EN ISO 16000-10; Emission test cell method
- EN ISO 16000-11; Sampling, storage of samples and preparation of test specimens
- ISO 16000-6; Measurement of VOCs in indoor and chamber air by sorbent tube and thermal desorption/ gas chromatography with FID/MS detection

EN ISO 16000-9 & 10

- 23˚C, 50%RH
- Requirements for control of conditions and *recovery of released chemical*
- Minimum requirement for sampling of chamber / cell after 3 and 28 days
- informative annex defining model room (17.4 m^3, 0.5 ach^{-1}) and area specific air flow rate for product types



ISO 16000-9

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3.15
volatile organic compound
VOC
organic compound that is emitted from the test specimen and all those detected in the chamber outlet air

NOTE 1 Due to practical reasons to be taken into account for test chambers, this definition differs from that defined in ISO 16000-6:2004. In ISO 16000-6, the definition is based on the boiling point range (50 °C to 100 °C) to (240 °C to 260 °C).

NOTE 2 The emission test method described in this part of ISO 16000 is optimum for the range of compounds specified by the definition of total volatile organic compounds (TVOC).

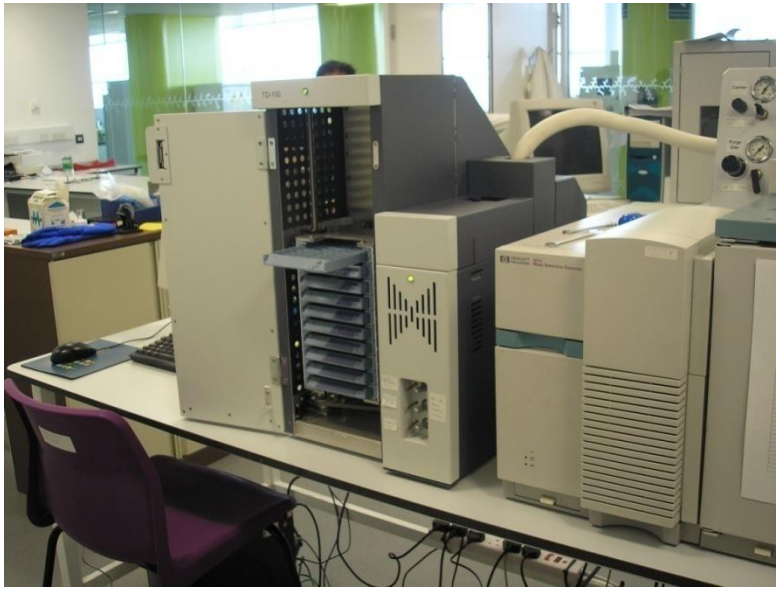
Recovery tests shall be performed in the test chamber using toluene and *n*-dodecane. Chamber concentrations shall be determined at 72 h after start of the test (i.e. first mandatory measurement, see 12.3). The mean recovery shall be greater than 80 % for toluene and *n*-dodecane. The results of recovery test shall be reported (concentration expected versus concentration measured).

NOTE 1 Low recovery of hygroscopic VOCs may occur in humidified air.

NOTE 2 Sink effects, leaks or poor calibration can cause difficulties to meet the minimum requirements. Sink and adsorption characteristics are very much dependent on the type of compound emitted. Additional recovery tests using target VOCs with different molecular weight and polarity can be used to increase understanding of these effects.

ISO 16000-6: (2004) revised 2011

- Measurement of VOC in indoor air and chamber air by pumped sampling using Tenax TA adsorbent tube and analysis by thermal desorption / gas chromatography with FID/MS detection
- Individual VOC (≥ 2 μgm^{-3})
- TVOC (FID/MS)(C6-C16)
- VVOC (<C6) and SVOC (>C16) outside TVOC range (informative)

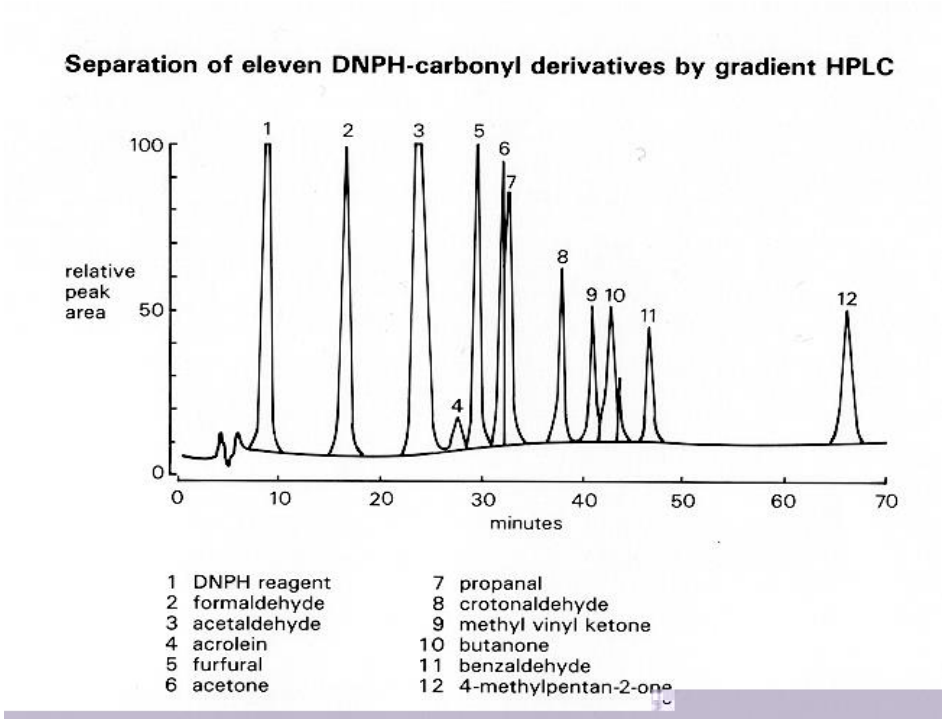


16000-6; performance

- The method is applicable to the measurement of non-polar and slightly polar VOCs..Using the principles specified in this method, some VVOC & SVOC can also be analysed (see A-D)
- Section 12 performance characteristics; requires uncertainty estimation including consideration of repeatability; ‘The accuracy and repeatability of the measuring method are important factors, which shall be determined….The accuracy of the VOC measurement method can be determined if atmospheres of known level can be reliably produced. This is relatively difficult and therefore most researchers only determine the repeatability of their measuring method by repeated sampling from a constant atmosphere.’

Other relevant EN and ISO standards on chamber air analysis

- EN ISO 16000-3 Measurement of aldehydes in indoor air (pumped)
- EN ISO 16017-1 Guidance on methods of VOC measurement in air (pumped)



Other published ISO standards

- 28 ISO standards published since 1994 on IAQ measurement, including 4 on testing VOC emission from in-vehicle materials and testing cabin air. Standards on sampling strategy for indoor air complement each methodology.
- Indoor air – Part 13: 2008 Determination of total (gas and particle-phase) polychlorinated dioxin-like biphenyls (PCBs) and polychlorinated dibenzo-p-dioxins/ dibenzofurans (PCDDs/PCDFs) – Collection on sorbent-backed filters
- Indoor air – Part 14: 2009 Determination of total (gas and particle-phase) polychlorinated dioxin-like PCBs and PCDDs/PCDFs – Extraction, clean-up and analysis by high-resolution gas chromatography/mass spectrometry

Other emission related standards

- BS ISO 16000-24:2009: Performance test for evaluating the reduction of VOC concentrations by sorptive building materials (except formaldehyde; which is BS ISO 16000-23:2009:)

- ISO 16000-25: 2011 Determination of the emission of semi-volatile organic compounds by building products and furnishing – Micro-chamber method

- ISO 16000-28:2012 Determination of odour emissions from building products using test chambers

ISO 16000 standards on under development

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- Indoor air -- Part 31: Measurement of flame retardants and plasticizers based on organophosphorus compounds - Phosphoric acid ester

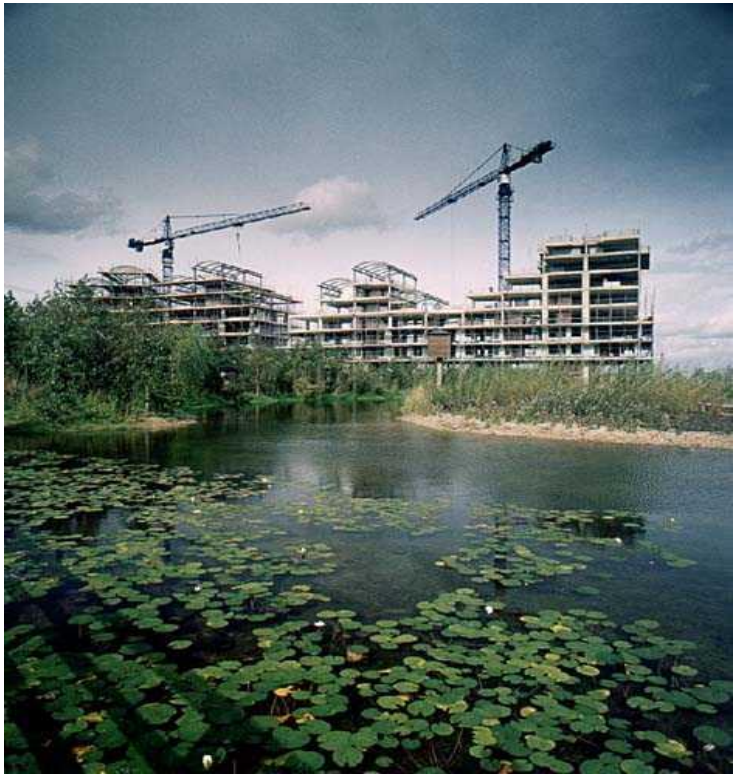
- Indoor air -- Part 33: Determination of phthalates with gas chromatography/mass spectrometry (GC/MS)

- Indoor air -- Part 35: Measurement of polybrominated diphenylether, hexabromocyclododecane and hexabromobenzene

(*probably not optimal for chamber testing as high volumes / flows*)

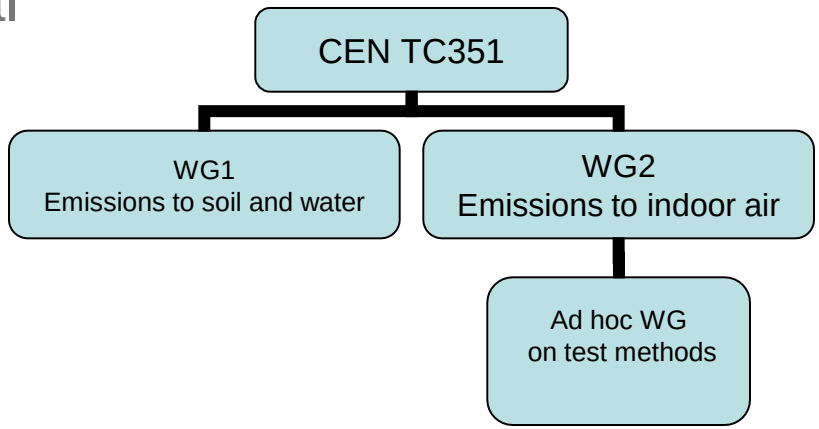
Construction Products Directive / Regulation

- 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
- Superceded by CPR, July 2013.



Mandate M/366 features

- Product emissions under normal conditions of use
- Test methods have horizontal character, wherever possible
- Methods validated; robustness and variability-uncertainty for each product group
- CEN standard committee TC 351 formed in April 2006
- Robustness testing prior to TS publication in 2013



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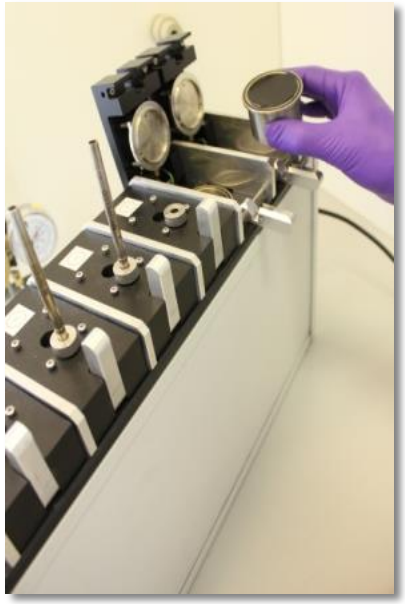
Harmonised standard

(currently voluntary Technical standard; CEN/TS 16516)

- Strongly based on the EN ISO standards but some differences and additional guidance and requirements
- Requires use of chamber method as reference method with some limits on chamber parameters (e.g. >20 L volume); intention to reduce interlaboratory variation
- Reference room (30 m^3) relating emission rate to concentration
- Recognises method can determine >C16 to C22
- Informative annex on VVOC (and SVOC)
- Recognises EN717-1 for testing formaldehyde emission from wood based products under existing regulations
- Enables use of indirect methods

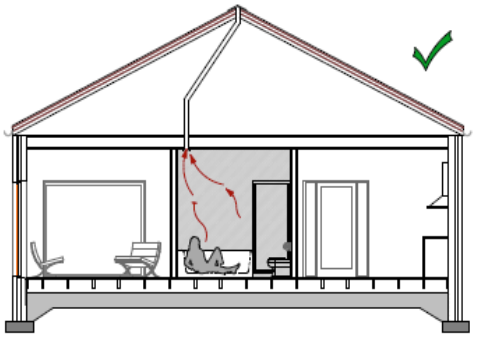
Indirect methods

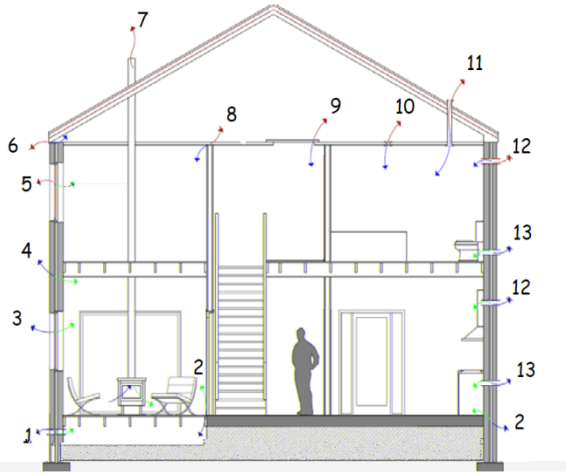
- Clause 11
- Simpler methods (easier/cheaper)
- Methods provide within field of application a result comparable or correlated to reference method
- Especially can be used for FPC
- Informative annexe gives examples: emission cell, microchambers

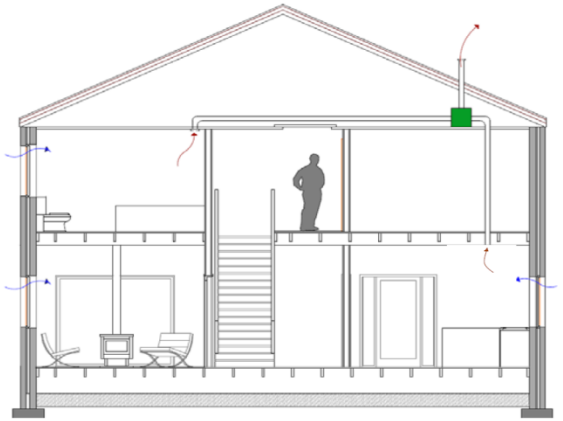


The CEN TS Reference Room

- Theoretical room that defines the exposure scenario
- Room dimensions:
  $30m^3$, 2.5m high, floor 4x3m
- 1 window and 1 door
- Room air exchange rate:
  constant 0.5 ach
- Temperature and humidity:
  23^oC, 50% RH







Reporting and Implementation

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Required reporting

Specific emission rates and concentration in the reference room air of:

- identified target VOCs (≥5 µg m^{-3})
- identified non-target VOCs (≥5 µg m^{-3})
- non identified VOCs (≥5 µg m^{-3} as toluene equivalent)
- carcinogenic substances (>1 µg m^{-3} '*as far as feasible*')
- TVOC & TSVOC (toluene equiv.)
- sum of identified non-target compounds and non-identified compounds;
- R value

Product evaluation

System of product evaluation to be defined (role of CEN, Commission and EGDS).

Classes likely, perhaps as currently used for formaldehyde release from wood based products;

- E1 <0.1 ppm in chamber test
- E2 >0.1 ppm.

TS 16516 quality control section 8.4

NOTE 1 Use of external reference materials spiked with VOCs with known emission rate, and with known emission decay profiles, are a useful tool for evaluating the performance of the whole procedure against primary standards, provided the quality of the reference materials is known. Determination of test chamber sink effects by recovery tests using target compound sources can be applied if suitable reference materials are not available, as described in EN ISO 16000-9.

NOTE 2 Participation in round robin tests and relevant independent analytical proficiency testing schemes are useful for comparing performance against a group of laboratories.

NOTE 3 Routine laboratory checks of analytical system performance can be conveniently carried out by spiking samplers (e.g. Tenax tubes) with a mix of compounds that is representative (in terms of analyte mass, polarity, volatility range, etc.) of the compounds of interests.

HSL, Sheffield have introduced a proficiency scheme with spiked tubes with VOCs of relevance to material emissions; assess performance of analysis

Certified primary gas standards; assess performance of sampling and analysis

Reference emission material; assess performance of full emission test procedure.

Some Issues

- Reference gases/ spiked tubes/ materials; what chemical range feasible, frequency of tests, acceptable costs,
- SVOCs; WHO / AgBB <C23 (380°C); how relate to exposure in air/surfaces/dust; ≥C23
- SVOC ambient chamber temperature air measurement or ISO elevated temperature (ISO 16000-25: 2011)
- Chamber recovery tests; just toluene and dodecane?
- Indoor chemistry – ignore?
- When is detection at 1 µg m^{-3} ‘feasible’; who decides
- And all those being raised by national committees currently commenting on the TS……

EN13999; Emission test of low solvent adhesives

- Part 1; Chamber test
- Part 2; VOC determination
- Part 3; volatile aldehyde determination
- Part 4; volatile diisocyanate determination

- Chamber >4 L
- Loading 0.4 m^2/m^3
- 0.5 ach
- Sample at 1, 2 and 10 days
- Determine specific emission rate; individual VOC and carcinogens
- Determine cumulative TVOC emission (1 to 10 days)

Resilient, textile and laminate floor coverings and paints

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- BS ISO 10580 – Test method for emission of VOCs
- Emission test chamber (20 – 1000 L)
- 23ºC 50%RH
- Determine specific emission rate (0.4 m^2/m^3 and 0.5-1ach)
- TVOC and individual VOC, plus formaldehyde (as 16000-6 and 16000-3)

EN 16402; 2013

- Paints and varnishes - Assessment of emissions of substances from coatings into indoor air - Sampling, conditioning and testing
- Mirrors TS 16516 closely
- Not covered by CPR
- Refers to pre-conditioning of samples (but not detailed description)

Main EU labelling schemes

Schemes

- AgBB, Germany (regulatory)
- M1, Finland (voluntary)
- DICL, Denmark (voluntary)
- AFSSET (voluntary) and Anses,(regulatory) France

Common features

- Chamber test of product to determine chemical emission rate
- Tests based on ISO 16000 (3, 6, 9, 10, 11) series standards
- Evaluate after 28 days (but some schemes have additional evaluation points)

Also many differences

e.g. TVOC, individual VOCs, formaldehyde, carcinogens, sensory test, LCi.

M1 scheme in Finland

- Uses chamber testing
- Evaluation only after 28 days
- Tests for TVOC, carcinogens, the irritants formaldehyde and ammonia
- Involves sensory evaluation for acceptability and odour intensity
- No pre-assessment of product composition, emissions after 3 days, single VOC (except carcinogens), unidentified VOC, SVOC.

DIBt / AgBB scheme - Germany

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- Assessment of construction products used internally according to Health aspects
- Step 1 registration and evaluation of ingredients
- Step 2 testing and evaluation of VOC and SVOC emissions
- Chamber tests with thresholds for TVOCSUM (target+non-target+non-identified+carcinogens), carcinogens, 'compounds of interest (LCI)' and other 'non-assessable' compounds
- Assessment 3 days and 28 days after product placed in chamber
- Notified regulation for flooring products.

French mandatory labelling

- DECREE relating to the labelling of construction and decoration products with their volatile pollutant emissions
- Labelling requirement:

  - on 1 January 2012 for products placed on the market;

  - on 1 September 2013 for all products.

TVOC at 28 days

<1.0 mg/m³ = A+

<1.5 mg/m³ = A

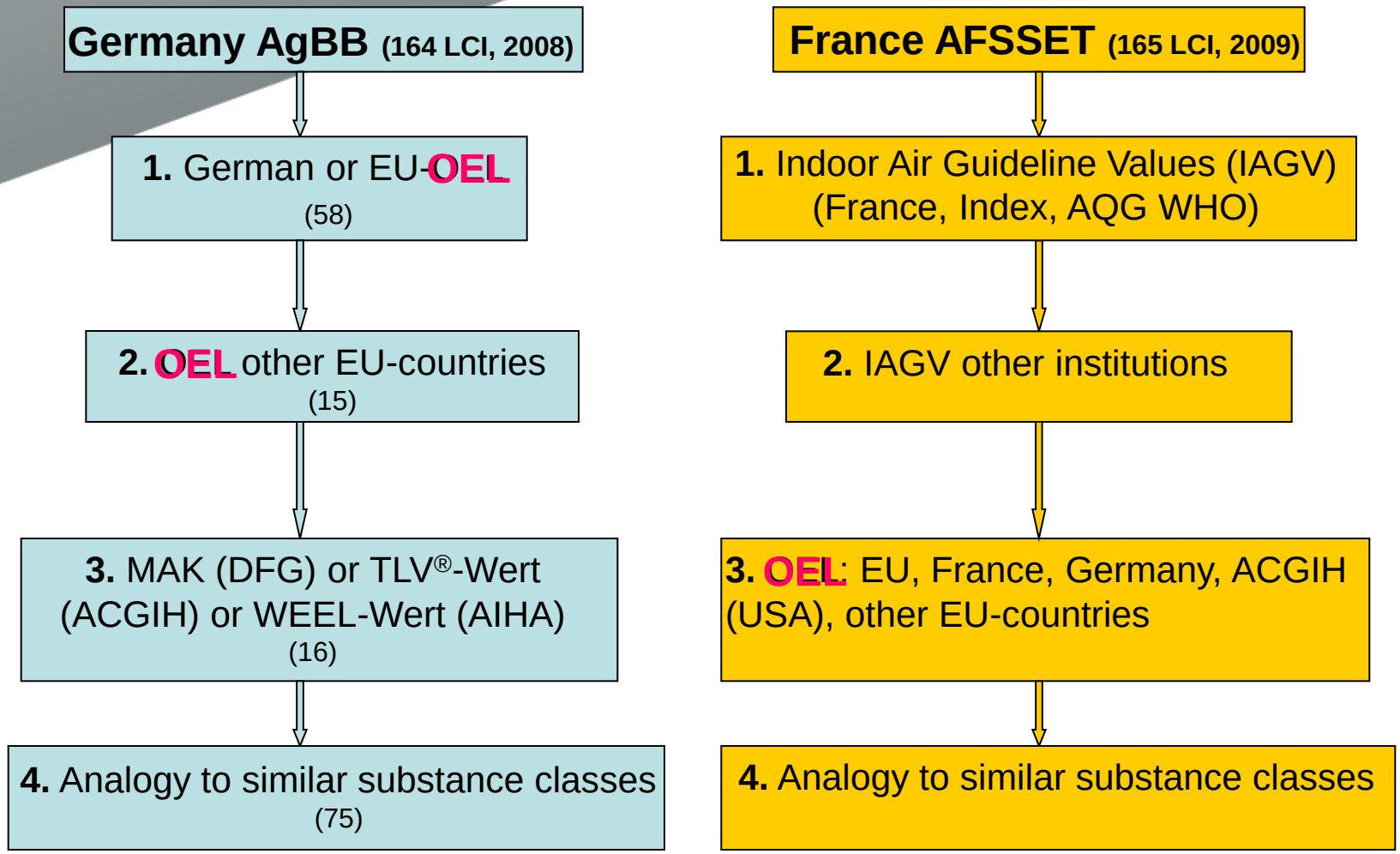
<2.0 mg/m³ = B

>2.0 mg/m³ = C

Carcinogens

10 individual VOCs **(LCI)**

Decision tree for choice of LCI setting basis



Examples of other labelling of low emitting products

- Blue Angel scheme includes furniture, computers, televisions.
- GUT scheme for carpets (industry scheme based in Germany)
- EMICODE for adhesives (industry scheme based in Germany)
- Nordic Swan e.g. flooring

Harmonisation of labelling

- In 2005 comparison of existing scheme (ECA report 25)
- ECA report no. 27 (2012); a road map to harmonisation.
- LCI harmonisation regarded as key part of harmonisation process as applied in Germany and France (plus stated intention in Belgium) and M1 and DICL schemes will include LCIs in future revisions.
- 2010; LCI task group established
- 2013 ECA report no. 29 recommends protocol for EU-LCI setting

EU-LCI* Definition

- EU-LCIs are health-based values used to evaluate emissions **after 28 days** from a single product during a laboratory test chamber procedure (as defined in the CEN TC 351 horizontal standard).
- EU-LCIs are applied in product safety assessment with the ultimate goal to avoid health risks from *long-term* exposure for the general population.
- They are intended only for evaluating emissions from building products and not for evaluating indoor air quality.

* '*Lowest Concentration of Interest*'

EU LCI Features

- Since primary emissions decline with time, this timescale (28 days) is considered to constitute a ‘worst case’ assumption for the long-term indoor air VOC emission scenario in the absence of oxidants.
- The test procedure (using chambers and correction factors relating to a ‘reference’ room) provides only an approximation to the situation in a real indoor environment; concentrations in actual rooms will depend on many factors including temperature, ventilation and the presence of other sources.

Protocol for the *de novo* derivation of EU-LCIs

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- A standard protocol for the transparent and harmonised derivation of EU-LCI values has been developed.
- The procedure consists of three main steps (ECA, 2013):
  - **data compilation,**
  - **data evaluation,** and
  - **derivation** of the EU-LCI on the basis of a standardised factsheet generated for each substance.
- Subject to necessary funding aim is to derive EU LCIs for all compounds included in current national regulations and future updates

Concluding remarks

- Controlling indoor sources of pollutants is an important strategy for controlling human exposure; especially when reducing rates of ventilation for energy savings
- CPD recognised the need for control of emission to indoor air 25 years ago
- ISO and EN standards have been available for ~10 years for emissions from construction products using environmental chamber tests (longer for formaldehyde from wood based).
- 'Voluntary' Emission Labelling schemes since ~1995 based on chamber testing and several regulatory national schemes

- 2013 proposed harmonised test method for construction product labelling under the CPR adapts EN/ISOs.

Concluding remarks

- Ensuring reproducible and accurate emission testing is an essential aspect of regulatory labelling of products; adoption of use of certified gases, participation in proficiency schemes and use of reference emission materials are tools for achieving and verifying a low uncertainty of measurement
- EU-LCI derivation is an integral part of the harmonisation framework for indoor products labelling schemes in the EU.
- A protocol for the transparent derivation of EU-LCI values has been prepared and applied to key indoor pollutants and hopefully the work will continue to encompass additional compounds (assuming funding is secured).

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