Hessian Agency for the Environment and Geology
Department I3 – Air Pollution, Emission

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The Question of Homogeneity inside a Chimney: Application of ISO 13528 to Stack Emission Proficiency Tests

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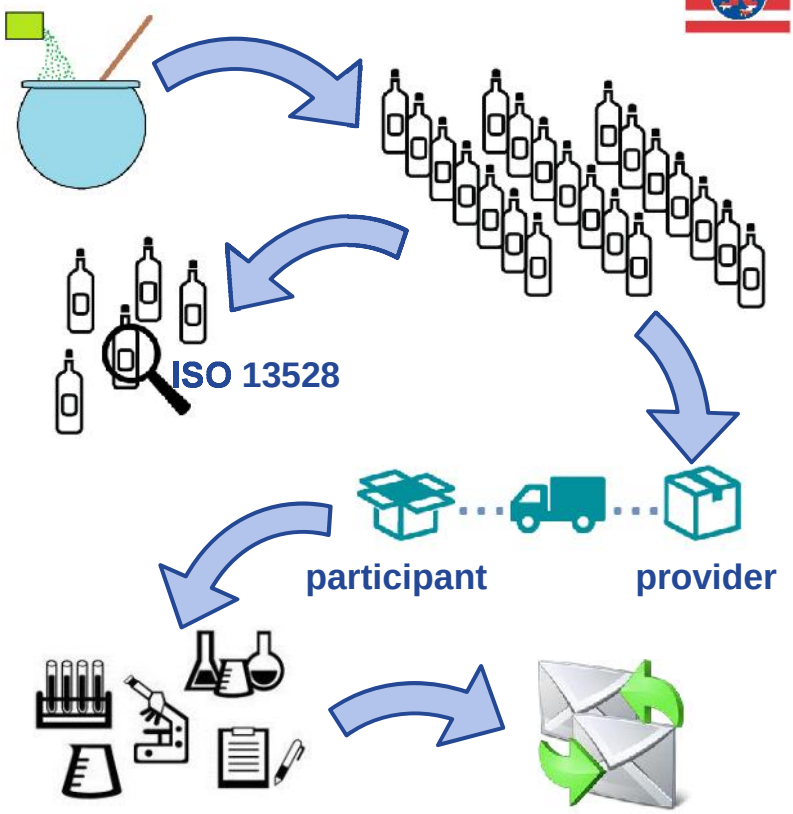
The „common“ Proficiency Test

- **Production** of a „huge“ batch of „test item“
- **Separation** into small (equal) portions
- **Homogeneity tests** (ISO 13528) on these small portions
- **Shipment** of test item portions to the participants
- **Participants analyze** the test item in their own laboratory
- **Results and evaluation**



Homogeneity of the „Test Items“: ISO 13528 Annex B

HESSEN

Procedure according to ISO 13528:

- Choose a property (e.g. a concentration)
- Choose a reliable laboratory (low repeatability standard deviation)
- Prepare and package the test items (ready for shipment)
- Select ≥ 10 items (randomly)
- Prepare ≥ 2 replicates of each of these items (repeat determination)
- Analyze all samples (in random order)
- Check relation of **between-samples standard deviation s_s** to **criterion for proficiency assessment σ_{pt}**:

$$s_s \leq 0.3\ \sigma_{pt}$$

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Stack Emissions and HLUG's Proficiency Test

HESSEN

- **Stack emissions:** generally exhaust gases from factories etc.
- **Important pollutants:** SO_2, NO_x, organic compounds, heavy metals (dust)
- **Measurement:** pollutant concentration and volume flow:

$$emission = concentration \times \frac{volume}{time}$$

A stack emission proficiency test therefore must include:

- volume flow measurement
- sampling
- analysis of the samples

→ **This requires an actual emission source! (preferably under controllable conditions)**

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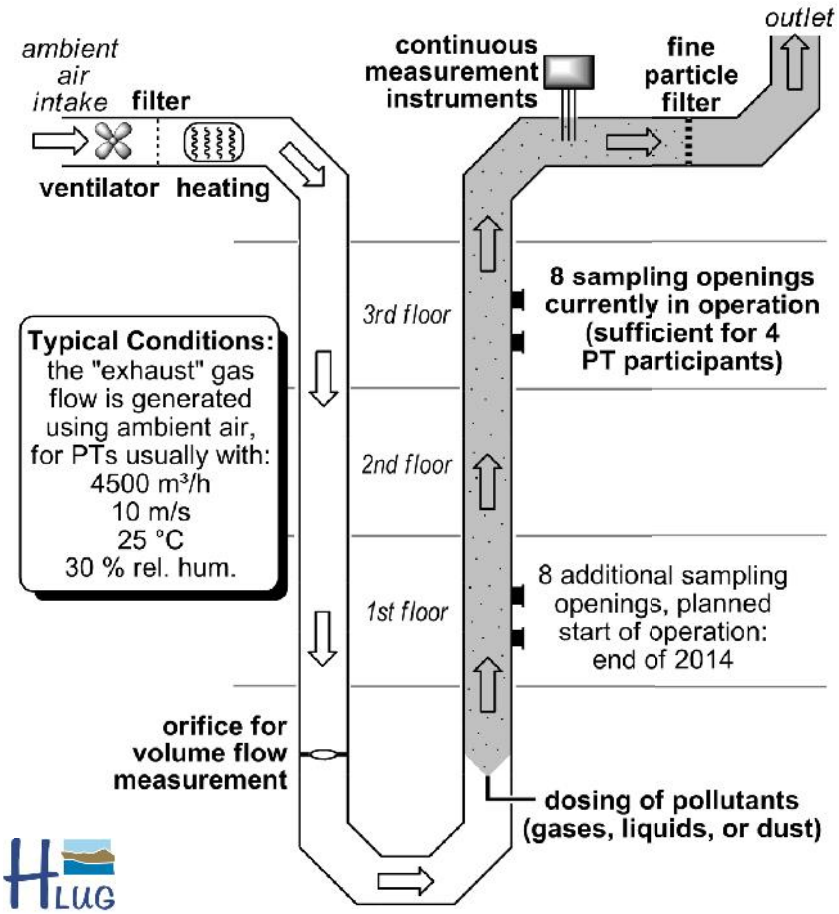
ESA – Emission Simulation Apparatus



Key figures:

- Total length: 110 m
- Height: ca. 30 m
- Inner diameter: 40 cm
- Artificial emission source with controllable conditions:
  - Volume flow
  - Temperature
  - Concentration
- Integrated into HLUG-building in Kassel, Germany

The „common“ Proficiency Test and HLUG’s Stack Emission Proficiency Test

„common” proficiency test:

- Production of a „huge“ **batch** of „test item“
- Separation into **small portions**
- Homogeneity tests on these **small portions** (concentrations may vary from portion to portion)
- **Shipment** of test item portions to the participants
- Participants analyze the test item in their own laboratory

HLUG:

- The „test item” (pollutant-doped air stream) is **produced constantly** during the proficiency test and **exists only for a few seconds**
- Samples are taken (as part of the PT) at **different positions** along the chimney
- Homogeneity tests on these **different positions** along the chimney (concentrations may vary from position to position)
- **Participants need to visit HLUG** in Kassel (Germany) to do the proficiency test
- Samples are taken by participants at HLUG’s ESA and are later analyzed in their own laboratory

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New Interpretation of Homogeneity in accordance with ISO 13528

HESSEN

Procedure according to ISO 13528:

- Choose a property (e.g. SO_2-concentration)
- Choose a reliable laboratory (HLUG)
- ~~Prepare and package the test items~~
- ~~Select ≥ 10 items~~
- ~~Prepare ≥ 2 replicates~~
- Analyze all samples
- Check relation of **between-samples standard deviation s_s** and **criterion for proficiency assessment σ_{pt}**:

$$s_s \leq 0.3\ \sigma_{pt}$$

Instead:

- Take ≥ 10 x 2 samples at different positions along the chimney

→ Equivalence of sampling positions (comparable conditions for all participants)

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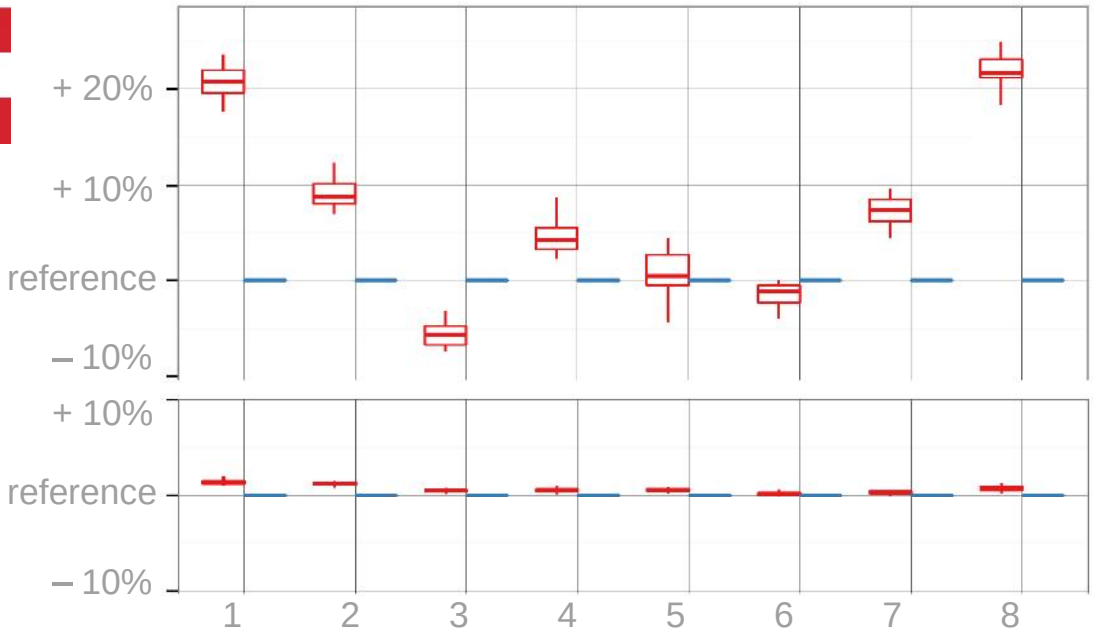
Gaseous Stack Emissions

HESSEN

- Test item: ***m*-xylene**-doted air
- Measurement: **total-C** with **FID** (Flame Ionization Detector)
- Preliminary results for 1st floor (red: sampling positions, blue: fixed reference):



Prior to dosing-optimization for measurements on 1st floor:

s_s = 9.9%

After optimization:

s_s = 0.30%

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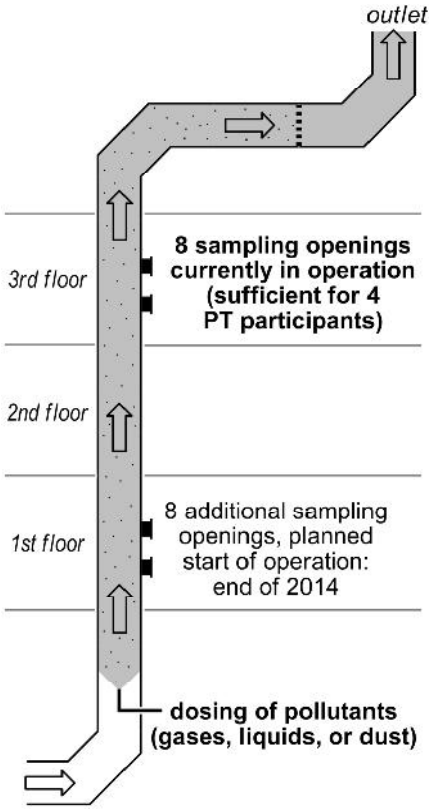
Gaseous Stack Emissions



Result of Homogeneity test (1st floor openings) in accordance with ISO 13528 Annex B:

- Determination of total-C (*m*-xylene) with FID, HLUG's current criterion for proficiency assessment: σ_{pt} **= 2.5%** (continuous measurement of total-C)
- **Before** dosing-optimization:
  s_s **= 9.9%** = 4.0 σ_{pt} (must be ≤ 0.3 σ_{pt})
  (but already homogeneous on 3rd floor!)
- **After** dosing-optimization:
  s_s **= 0.30%** = 0.12 σ_{pt} (must be ≤ 0.3 σ_{pt})
  (suitable for proficiency tests)

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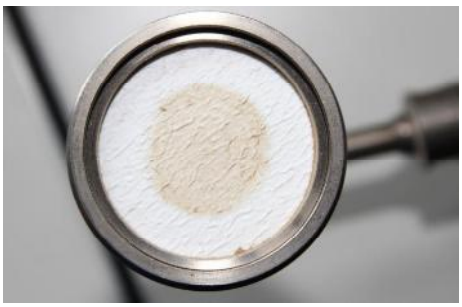
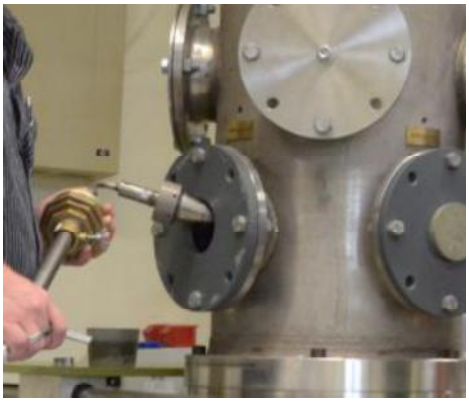
Particulate Stack Emissions

Additional Problems compared to gases and vapours:

- Homogeneity is much more difficult to achieve: Particles show **size-dependent inertia**
- The measured property is **mass per volume**, meaning: dust must be collected and weighed
- Discontinuous measurement is necessary: **30 minute sampling** (using a weighed filter), taken at 4 points along cross section (grid measurement, each for 7.5 min)





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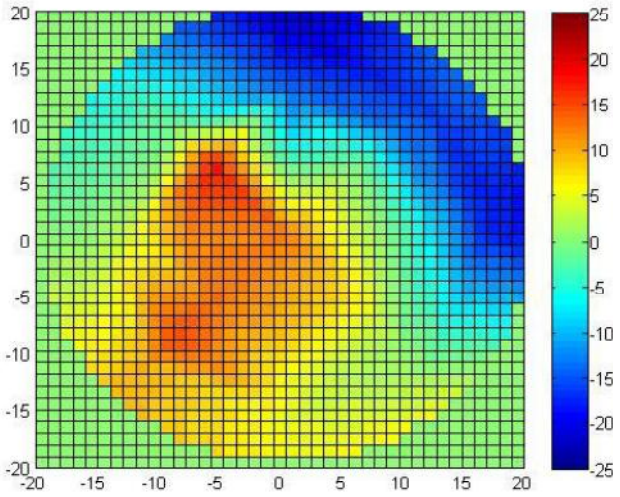
Particulate Stack Emissions

HESSEN

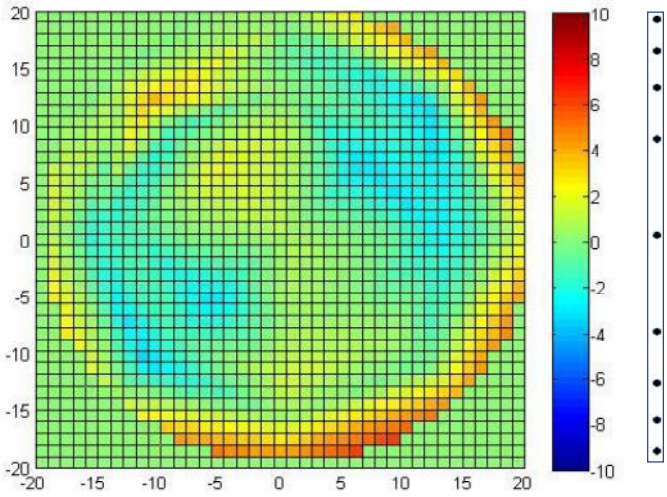
Dosing-optimization for measurements on 1st floor:

- Continuous measurements of fine particle concentration at 33 points along cross section (gives only relative values)
- Optimization progress so far (please note the different scales!):

Initial situation:



Result after "some" improvements:



(But again: homogeneous on 3rd floor already!)

Conclusion

HESSEN

- Stack emission measurements require determination of both concentration and volume flow
- HLUG offers stack emission proficiency tests on a factory chimney replica (ESA)
- Participants need to come to the ESA for proficiency testing
- The "test item" (pollutant-doped air) is generated constantly and exists only for a few seconds (from dosing-lab to roof)
- Comparable conditions for all participants means here: equivalent sampling positions along the chimney
- This requires homogeneous distribution of pollutants inside the chimney
- The equivalence of sampling openings along HLUG's ESA could be assessed by (analogue) application of ISO 13528 Annex B
- Sufficient homogeneity was demonstrated for gases and liquid vapors, similar assessments for dust are in progress