

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

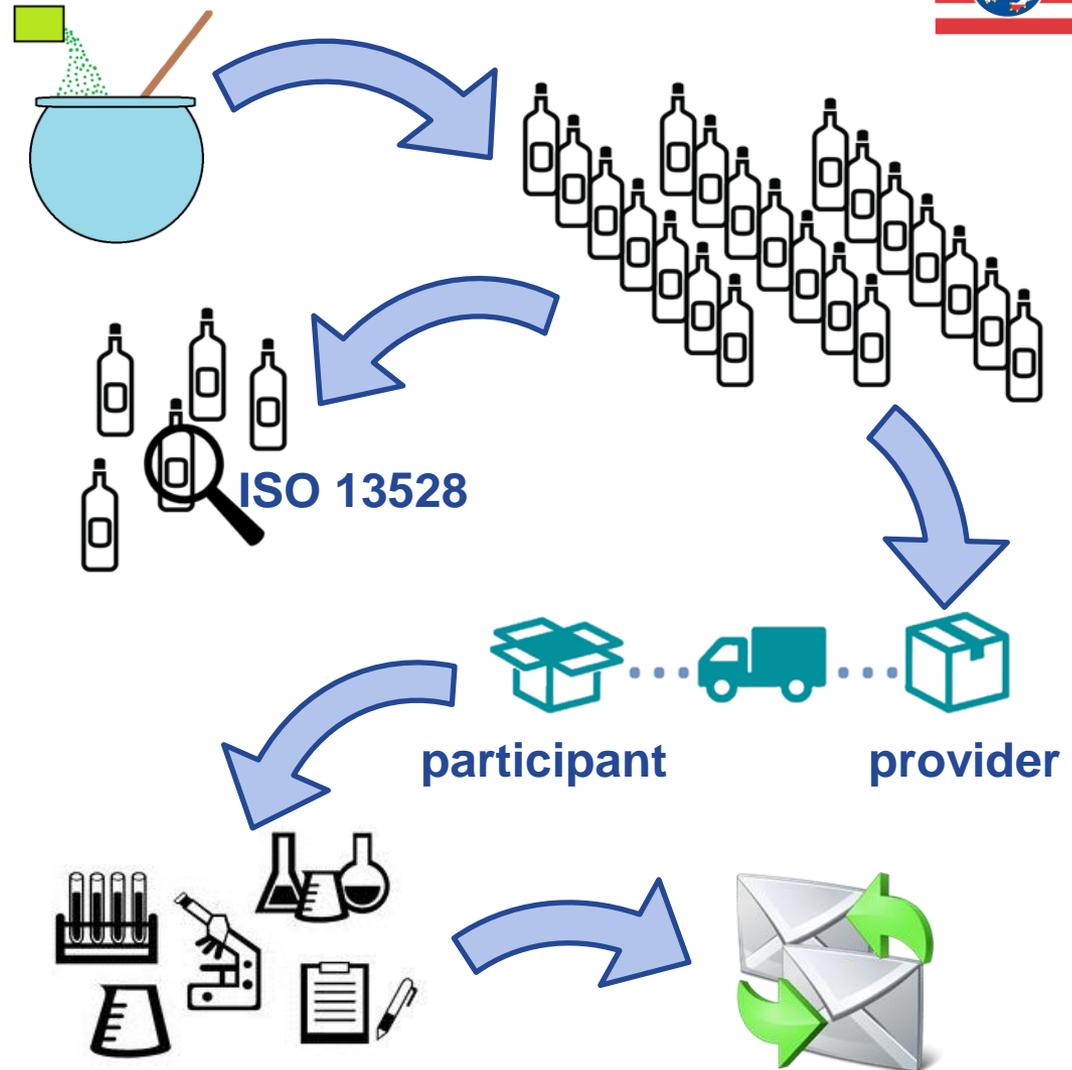
8<sup>th</sup> EURACHEM PT Workshop, Berlin, 9<sup>th</sup> October 2014





# 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

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  $\geq 10$  items (randomly)
- Prepare  $\geq 2$  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  $\sigma_{pt}$** :

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

# Stack Emissions and HLUG's Proficiency Test

- **Stack emissions:** generally exhaust gases from factories etc.
- **Important pollutants:** SO<sub>2</sub>, NO<sub>x</sub>, organic compounds, heavy metals (dust)
- **Measurement:** pollutant concentration and volume flow:

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

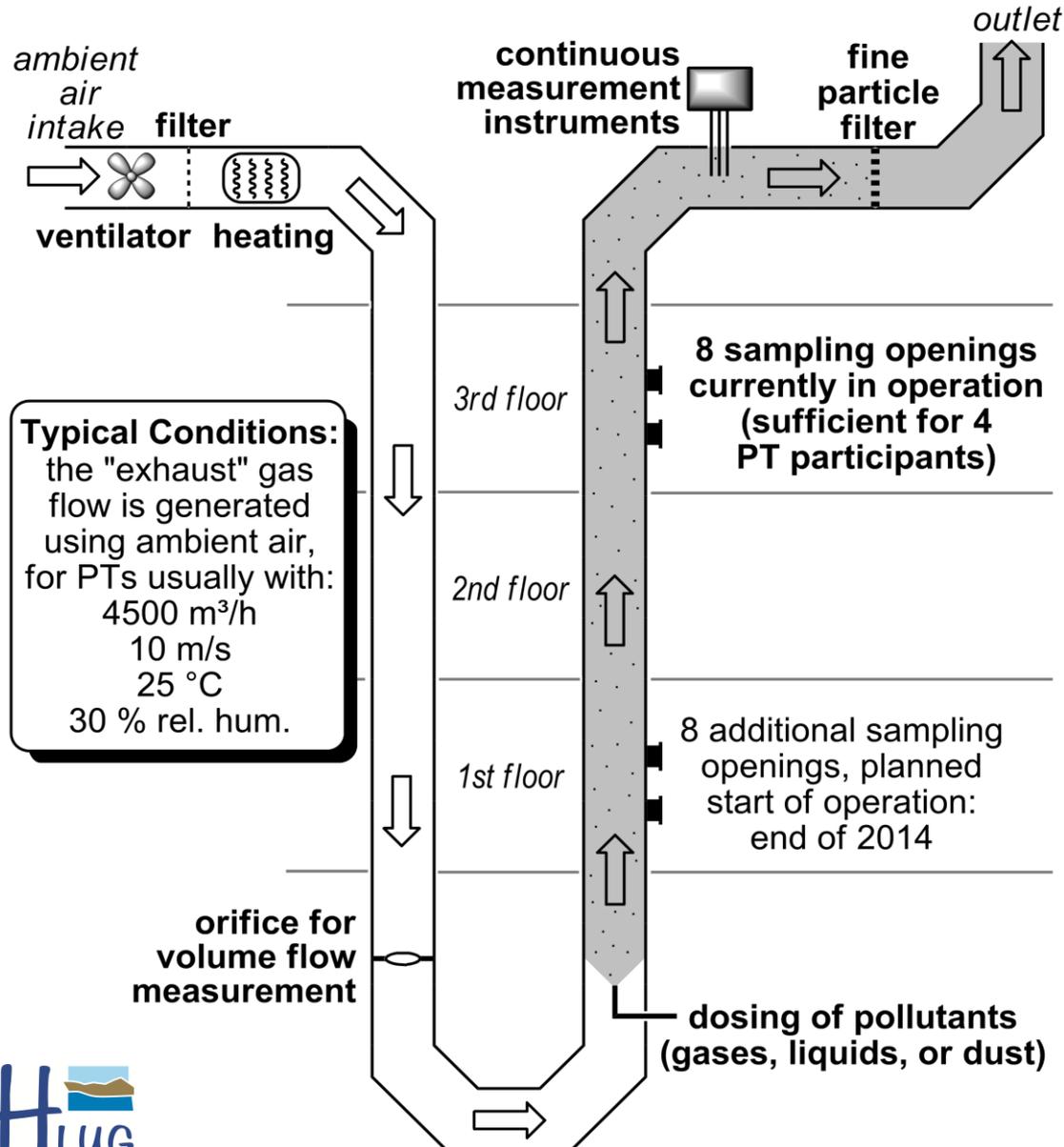
**A stack emission proficiency test therefore must include:**

- volume flow measurement
- sampling
- analysis of the samples



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

# 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

# New Interpretation of Homogeneity in accordance with ISO 13528

Procedure according to ISO 13528:

- Choose a property (e.g. SO<sub>2</sub>-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  $\sigma_{pt}$** :

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

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

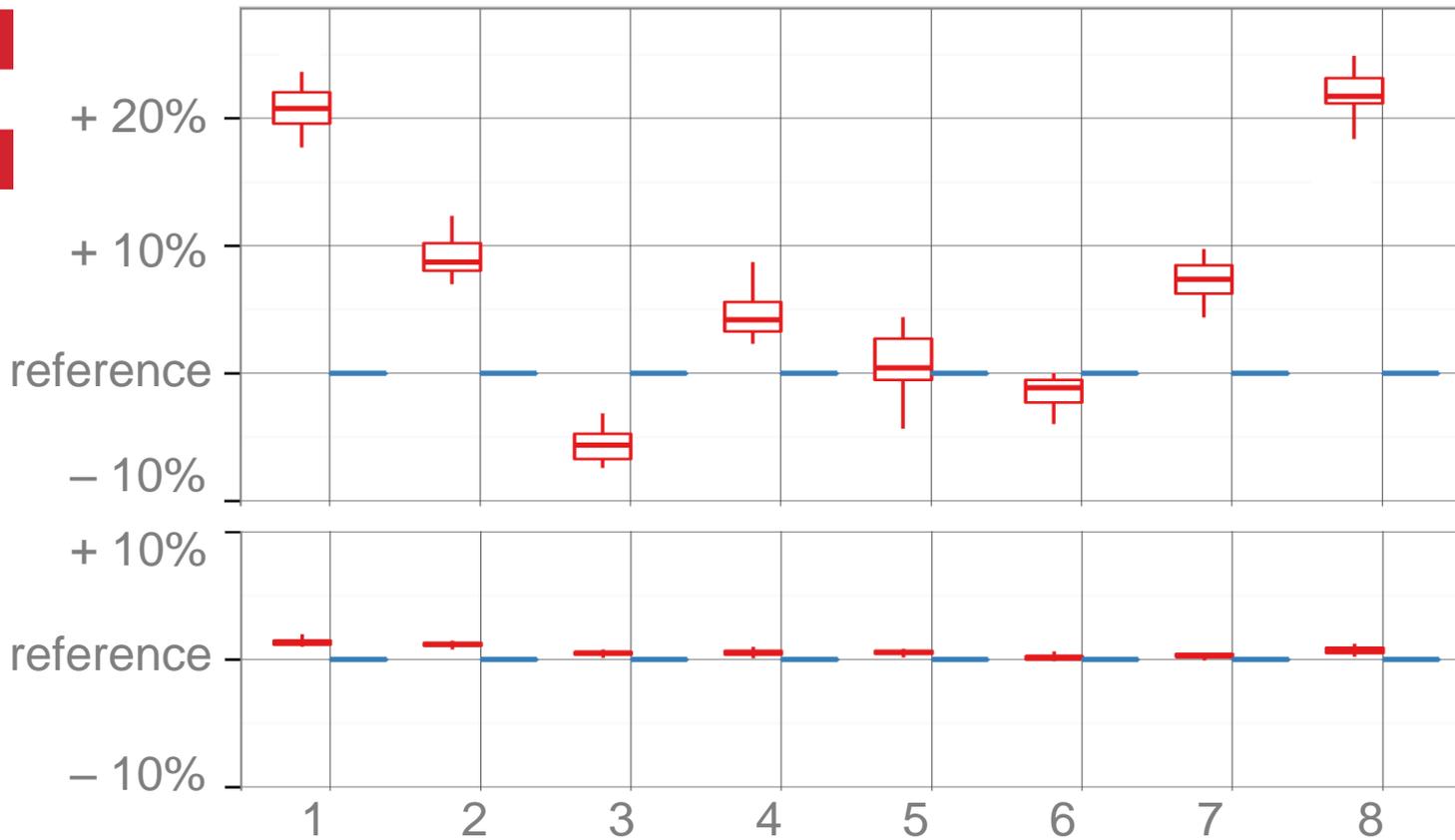
***Instead:***

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



# Gaseous Stack Emissions

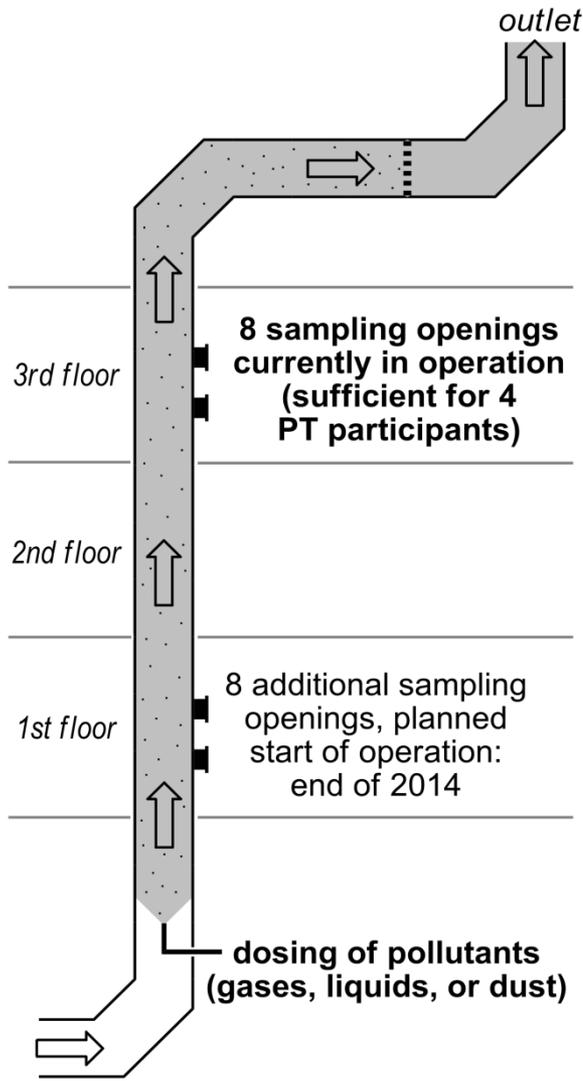
- Test item: *m*-xylene-doted air
- Measurement: **total-C** with **FID** (Flame Ionization Detector)
- Preliminary results for 1<sup>st</sup> floor (red: sampling positions, blue: fixed reference):



Prior to dosing-optimization for measurements on 1<sup>st</sup> floor:  
 $s_s = 9.9\%$

After optimization:  
 $s_s = 0.30\%$

# Gaseous Stack Emissions



## Result of Homogeneity test (1<sup>st</sup> floor openings) in accordance with ISO 13528 Annex B:

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

# 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)

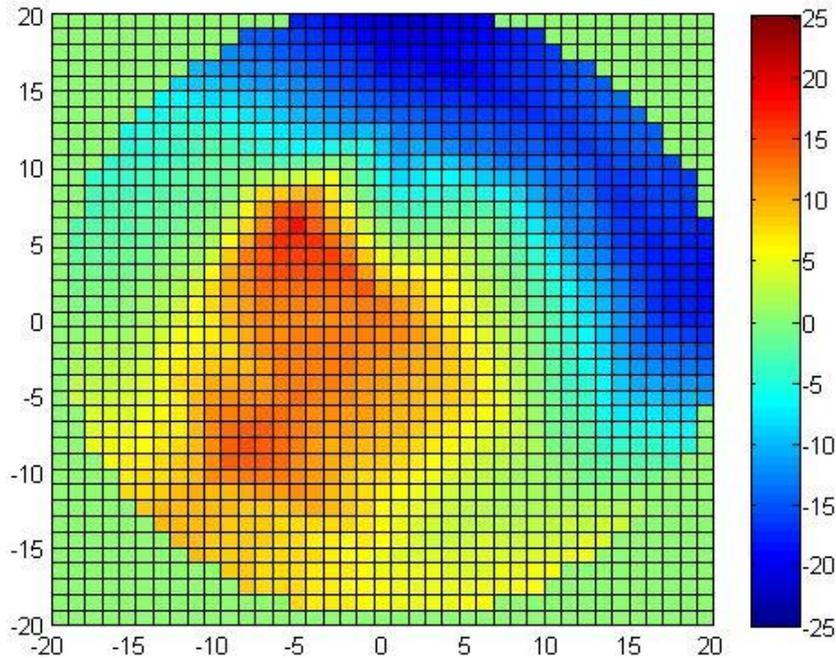


# Particulate Stack Emissions

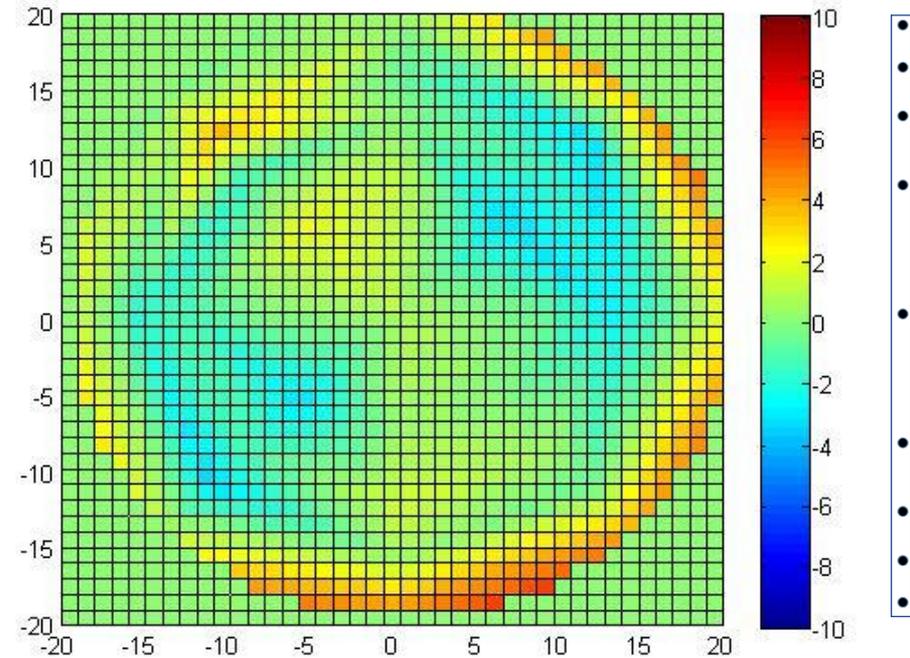
## Dosing-optimization for measurements on 1<sup>st</sup> floor:

- 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 3<sup>rd</sup> floor already!)*



# Conclusion

- 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