ISO/TC 158 Analysis of gases

GAS2015

NEN

On Development and Characterisation of a Portable Standard Gas Generator

Daiana Leuenberger, Céline Pascale, Andreas Ackermann, Myriam Guillevic, Bernhard Niederhauser

Federal Institute for Metrology METAS

The EMRP JRP ENV55 **MetNH3 (Metrology for Ammonia in Ambient Air) aims at providing certified reference material (CRM) in relevant Ammonia (NH_3) amount fractions (0.5-500 nmol/mol)** for air quality monitoring.

For this purpose and as alternative to CRM in gas cylinders, the Federal Institute of Metrology **METAS is developing a traceable portable gas standard generator**. This device is based on the specific **temperature dependent permeation** of the reference substance (NH_3) through a membrane into a flow of carrier gas (N_2 or air). This mixture is then diluted to desired amount fractions in 1 to 2 steps according to $x_p = x_B \frac{\dot{V}_1}{\dot{V}_1+\dot{V}_2} + x_{DG}$ (where x_P (nmol/mol) is the resulting NH_3 concentration, x_B the primary NH_3 concentration, $\dot{V}_1$ the base gas flow, $\dot{V}_2$ the dilution gas flow, x_{DG} the background concentration of ammonia in the dilution gas).

Second dilution step and traceability of all components are novelties compared to commercially available gas generators.
Here we present **first insights into the development** of said instrument, the **characterisation of individual components** leading to the **uncertainty estimation for the generated NH_3 gas mixture** according to GUM, which is aimed to be **<3 %**.

Moreover, we present **first results of the study on adsorption/desorption effects** of NH_3 on different tubing materials.

Permeation

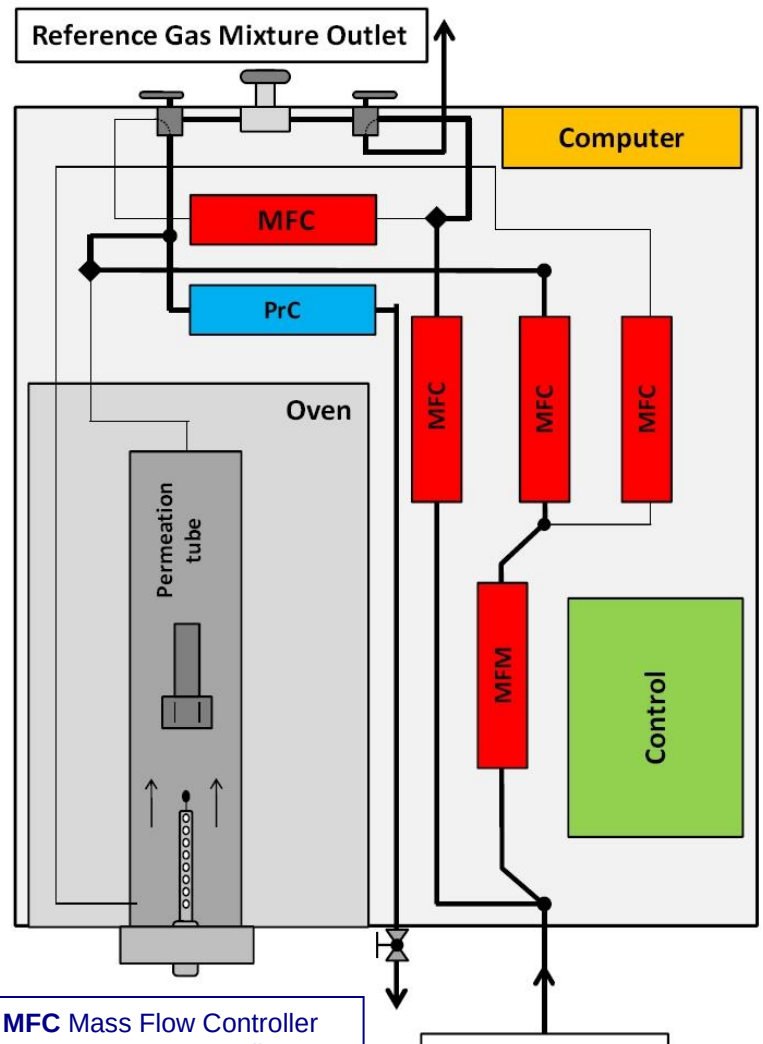
The controlled permeation of a substance through a polymer membrane is **primarily temperature dependent** and results in a **measureable mass loss over time**.
A **permeation tube** with a traceable **permeation rate** $Pr = \frac{\Delta m}{\Delta t}$ (SI-traceably determined by a Magnetic Suspension Balance (MSB), resolution= 10^{-6} g) is placed in the **permeation oven**.

Long-term expanded measurement uncertainty:
$Pr = < 1.7\ \%$

Contribution to total measurement uncertainty:
$Pr = \ 20\% - 80\ \%$

Surface Coating

Surfaces in contact with NH_3 have been coated with commercially available **SilcoNert 2000** (SilcoTek). This **significantly reduces adsorption losses and stabilisation time** (see below).



MFC Mass Flow Controller
PrC Pressure Controller
MFM Mass Flow Meter

Dilution

First dilution step qv_1: Reduces NH_3 amount fractions generated in permeation oven to (> 100 nmol/mol), expanded measurement uncertainty (k=2) $qv_1 = 0.2\ \%$

Split flow qv_3: Subdivision, expanded measurement uncertainty (k=2) $qv_3 = 0.3\ \%$

Second dilution step qv_4: Reduces NH_3 amount fractions generated in permeation oven to (> 0.5 nmol/mol) expanded measurement uncertainty (k=2) $qv_3 = 0.2\ \%$

Contribution of dilution steps to total measurement uncertainty:
$qv_{tot} = < 30\ \%$

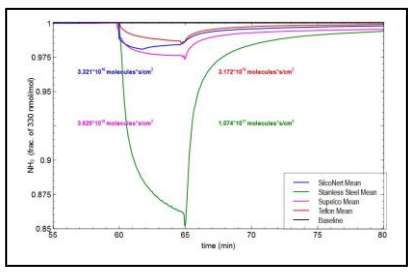
Additional Contributions

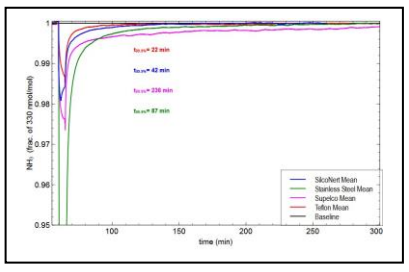
Purity of the matrix gas: Increasing with decreasing NH_3 amount fractions (0.05 ± 0.05 nmol/mol), significantly increases total measurement uncertainty by up to 100%.

Temperature regulation in the oven: Instability increasing with increasing temperatures, corresponding to an increase in the uncertainty of 2% for a difference in T= 0.1 K.

Reduction of adsorption losses and stabilisation time by surface coating

Following the approach of ***Vaittinen (2013)***, 4 different types of tubing (**Stainless Steel 316**, Polymer (**PFA**), 2 fused silica coatings applied to Stainless Steel 316 (**Supelco**, **SilcoNert 2000**)) have been exposed to a gas mixture of **330 ppb NH_3 in N_2** (Alphagaz II). **Adsorption loss** (molecules*s/cm^2) and **stabilisation time** ($t_{99.9\%}$) have been evaluated.





	t (99.9%)	adsorbed mol*s/cm^2 ($t_{99.9\%}$)
PFA	22 min	$3.172 * 10^{16}$
SilcoNert 2000	42 min	$3.321 * 10^{16}$
Stainless Steel 316	87 min	$1.074 * 10^{17}$
Supelco	236 min	$3.629 * 10^{16}$

We recommend to have SilcoNert 2000 (SilcoTek) applied to Stainless Steel surfaces in contact with NH_3.

MetNH3

METAS

EMRP
European Metrology Research Programme
Programme of EURAMET

The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union

Reference:
Vaittinen et al.: Adsorption of Ammonia on stainless steel and treated polymer surfaces. Applied Physics B, 2013.

Contact details:
Daiana Leuenberger
daiana.leuenberger@metas.ch
www.metas.ch
http://metnh3.eu