Calibration of diffusive samplers for nitrogen dioxide using the reference method – Evaluation of measurement uncertainty

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Abstract For many years, diffusive samplers (modified Palmes tubes) for nitrogen dioxide (NO_2) have been used in the North Rhine-Westphalia air quality network. Due to a large upgrade of automated NO_x measuring systems with monitors representing the reference method a comprehensive re-validation of the diffusive method was performed. The uptake rate of the samplers was evaluated by comparison with the reference method using 286 data pairs (approx. one month exposure) from nine different monitoring stations over three years (2007 to 2009). The uptake rate determined is 0.734 ± 0.004 cm³/min. This value is fully in-line with theoretical estimations. Evaluations according to the "Guide to the demonstration of equivalence of ambient air monitoring methods" reveal an expanded uncertainty of 20.0% for a single value (monthly average). The expanded uncertainty of annual means is found to be 12.6%. Measurements of NO_2 with diffusive samplers can therefore meet the data quality objectives for fixed measurements according to Annex I of the European Directive 2008/50/EC.

Kalibrierung von Passivsammlern für Stickstoffdioxid mithilfe des Referenzverfahrens – Ermittlung der Messunsicherheit

Zusammenfassung Im Luftqualitätsmessnetz LUQS des Landes Nordrhein-Westfalen werden seit mehreren Jahren Passivsammler (modifizierte Palmes-Röhrchen) zur Messung von Stickstoffdioxid (NO_2) eingesetzt. Aufgrund des Austauschs der NO_x-Messgeräte im kontinuierlichen Messnetz gegen neue Geräte, die das Referenzverfahren repräsentieren, wurde eine umfassende Neu-Validierung der Passivsammlermethode durchgeführt. Die Aufnahmerate der Sammler wurde auf der Basis von 286 Datensätzen berechnet, die im Laufe von drei Jahren (2007 bis 2009) an neun verschiedenen Messstationen durch Parallelmessungen mit dem Referenzverfahren über jeweils ca. einen Monat gewonnen wurden. Die experimentell ermittelte Aufnahmerate beträgt 0,734 ± 0,004 cm³/min und steht in sehr guter Übereinstimmung mit theoretischen Berechnungen. Auswertungen auf der Basis des Leitfadens zur Äquivalenzprüfung von Messverfahren ergaben eine erweiterte Messunsicherheit von 20,0 % für einen Einzelwert (Monatsmittelwert). Die erweiterte Messunsicherheit von Jahresmittelwerten beträgt 12,6 %. Messungen von NO_2 mit Passivsammlern können somit die Datenqualitätsziele für ortsfeste Messungen gemäß Anhang I der europäischen Richtlinie 2008/50/EG erfüllen.

1 Introduction

Implementing the Directive 2008/50/EC of the European Parliament and the Council of 21 May 2008 on ambient air quality and cleaner air for Europe [1] there is a considerable need of nitrogen dioxide measurements, especially near roads with heavy traffic. It is hardly possible to accommodate this demand by continuous measurements only. Consequently, diffusive samplers can partially substitute and supplement fixed monitoring as an instrument for the assessment of air quality, provided that they fulfil the specific data quality objectives given in the Directive. This demonstration of equivalence can be performed by calibrating the diffusive samplers with the reference method.

2 The Reference Method

In 2005 the new CEN standard EN 14211:2005 [2] was published. The application of this standard is mandatory for ambient air monitoring according to the European Directive mentioned above.

The standard EN 14211 contains stringent requirements for type approval and routine monitoring in networks. According to expert judgement, most automated monitoring systems (AMS) on the market in 2005 were not able to meet the requirements of this standard. This also held true for the AMS Environnement AC 31 M used in the LUQS network in North Rhine-Westphalia at that time. Therefore, all notable manufacturers developed new types of instruments and underwent type approval procedures. This was also the case for the instrument Environnement AC 32 M.

A weak point of the old instrument AC 31 M was a significant cross-sensitivity against water vapour (air humidity). The new AMS AC 32 M uses an integrated sample dryer and meets the requirements of the new standard also in this issue. In field measurements, the cross-sensitivity of the old type of instrument (AC 31 M) could induce an underestimation of real NO_x concentrations of approximately 10% and more.

Annex VI D of the Directive 2008/50/EC explicitly says: *"All equipment used in fixed measurements must comply with the reference method or equivalent by 11 June 2013"*.

Starting in the beginning of 2007 the new instrument Environnement AC 32 M was introduced to the LUQS network. For various reasons a complete exchange of a whole generation of AMS can only be performed within several years. As a consequence, this leads to a step-by-step increase of measured NO_x concentrations which reflects the real situation.

3 Use of diffusive samplers

Facing the increasing need of NO_2-measurements in heavily trafficked streets North Rhine-Westphalia State Agency for Nature, Environment, and Consumer Protection (LANUV; formerly LUA) performed a comprehensive validation campaign in 2004 to 2005 for a specific type of diffusive samplers [3]. The uptake rate of these samplers – a Palmes type tube of Passam AG (Männedorf, Switzerland) modified with a glass

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**Comparison of Annual Averages 2006
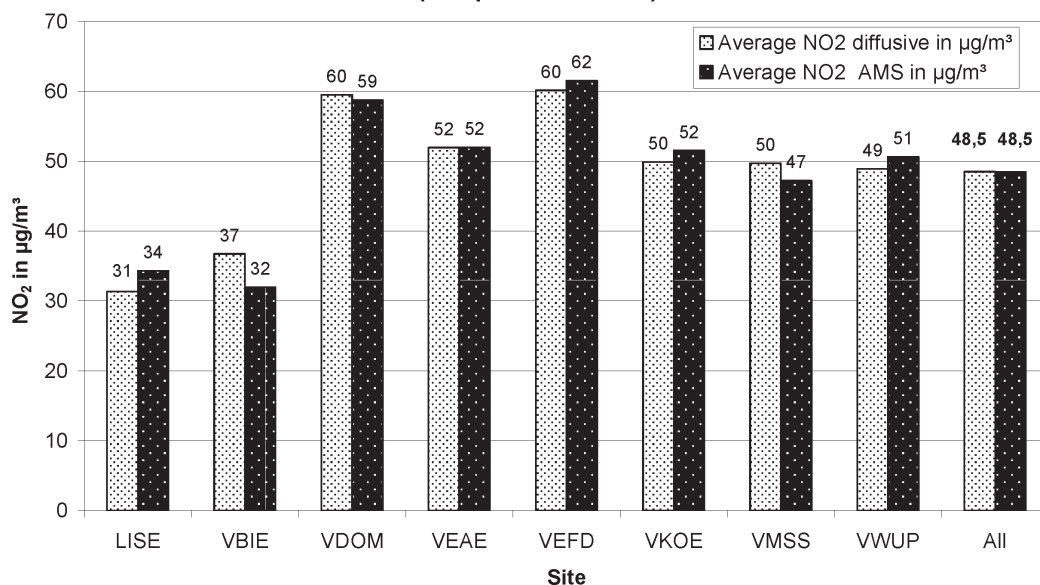(Independent Period)**

Figure 1. Annual averages for NO_2 measured with AMS and diffusive samplers.

frit as turbulence barrier – was determined at ten different monitoring sites over 14 months. This uptake rate of 0.827 cm³/min was used in the whole network of North Rhine-Westphalia until end of 2008. Details of this technique and the validation can be found in [4].

For the first time, the year 2006 represented a comparison period completely independent of the validation experiments. **Figure 1** shows NO_2 annual averages from eight monitoring stations measured with AMS and with diffusive samplers [5].

Differences between individual averages measured continuously and with diffusive samplers are generally less than 10%. For the average of all stations results are exactly identical. These and further results led to the conclusion that annual averages measured with diffusive samplers can meet the data quality objective of Directive 2008/50/EC even for fixed measurements [5].

Also in the following years the validity of the calibration of the diffusive samplers was continuously checked at various stations in the framework of ongoing QA/QC procedures. In the years 2007 to 2009 increasing differences between the AMS results and those of the diffusion tubes were observed with the AMS data giving higher concentrations. Comprehensive and rigorous checks of all procedures involved showed clearly that these differences were definitely due to the exchange of AMS with an increasing use of the new instrument AC 32 M [6; 7].

Consequently, at the end of 2009 the uptake rate of the diffusive samplers was newly determined based on results of 2007 to 2009 with the new AMS AC 32 M. This uptake rate is used for all data since 2009. In this way, a direct link is created between the diffusive sampler method and the reference method laid down in Directive 2008/50/EC.

3.1 Determination of the uptake rate for modified Passam tubes

The uptake rate was determined by parallel measurements of the diffusive method with the reference method according to EN 14211 at nine monitoring stations over three years (2007 to 2009). The evaluation is based on 143 exposure intervals with duplicate samples for the diffusive method so that 286 data pairs exist. The exposure interval was approximately four weeks/one month.

In passive samplers molecules diffuse because of a concentration gradient through an intake opening with the cross-section A along a diffusion path with the length l to a sampling medium by which they are adsorbed. This process is described by Fick's first diffusion law:

$$\frac{\mathrm{d}m}{\mathrm{d}t} = D \cdot \frac{A}{l} \cdot \mathrm{d}c \qquad (1)$$

m_i mass in pg
t exposure period in min
A cross section of sampler in cm²
l length of diffusion path in cm
D diffusion coefficient in cm²/min
c concentration in µg/m³

Assuming that the concentration at the intake opening equals the ambient concentration c, while the concentration at the surface of the sampling medium is zero, then the adsorbed amount of substance is proportional to the concentration c and the exposure time t:

$$m = D \cdot \frac{A}{l} \cdot c \cdot t \qquad (2)$$

The parameter

$$UR = D \cdot \frac{A}{l} \quad \text{in cm³/min} \qquad (3)$$

is known as the uptake rate of the sampler with regard to the substance i. The unit cm³/min may be considered to be a reduction of the unit pg · (µg/m³)$^{-1}$ · min^{-1}.

This simplifies eq. (2) leading to eq. (4):

$$m = UR \cdot c \cdot t \qquad (4)$$

More details on the principles of measurements with diffusive samplers may be found in EN 13528-3 [3].

The uptake rate can be determined experimentally by plotting the dose of NO_2 (product of average concentration

Uptake Rate (Data Capture > 75%) for AMS AC 32 M (Environnement)
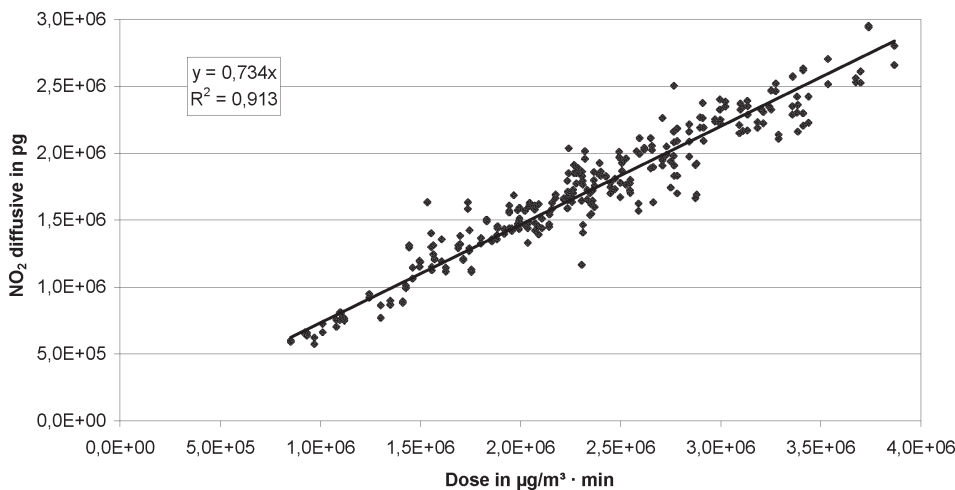**9 stations (2007 - 2009) - 286 data pairs**

$y = 0{,}734x$
$R^2 = 0{,}913$

Figure 2. Experimental determination of the uptake rate ($n = 286$).

measured with the reference method and the exposure time of the sampler) on the x-axis and the mass of analysed NO_2 from the sampler on the y-axis. The slope of the regression line is the uptake rate (**Figure 2**).

The uptake rate (rounded) is $UR = 0.734 \pm 0.004$ cm³/min. The regression line was forced through the origin because of the following considerations:

● No significant field blanks for the diffusive samplers were observed.

● The time coverage of the reference method is less than 100% due to calibrations, maintenance and other technical reasons. This may cause an artificial bias which does not exist in reality.

● When calculating the regression with an intercept the values were found to be below the detection limit of the method.

The results for the reference method are related to 20 °C as required by directive 2008/50/EC. Therefore, also this uptake rate corresponds to this temperature. Variations of temperature during the measurement periods are reflected in the uncertainty budget of the uptake rate.

3.2 Theoretical assessment of the uptake rate

In order to check the plausibility of the experimentally determined uptake rate a theoretical estimation was done as follows [8]:
Equation (3) may be modified as

$$UR = D/R \tag{5}$$

with $R = l/A$ (resistance) $\tag{6}$

If the diffusion path consists of two sections, namely
section (1) = air and
section (2) = glass frit
the total resistance may be calculated as:

$$R = l_1/A_1 + l_{2\text{eff}}/A_{2\text{eff}} \tag{7}$$

with
$l_{2\text{eff}} = l_2 \cdot \tau$ ($l_{2\text{eff}}$ = effective length)
$A_{2\text{eff}} = A_2 \cdot f$ ($A_{2\text{eff}}$ = effective cross section)

This leads to:

$$R = l_1/A_1 + (l_2 \cdot \tau)/(A_2 \cdot f) \tag{8}$$

f is the porosity and describes the fraction of void space in a material, in the present case the fraction of air in the glass frit related to the bulk volume of the frit [9]. For the glass frits used in this study $f = 0.33$.

τ is the so-called tortuosity and is the ratio of the real path length of a molecule through the frit and the thickness of the frit [9; 10]. τ may be estimated from f using the equation:

$$\tau = 2.23 - 1.13 \cdot f \tag{9}$$

Figure 3 shows a sketch of the modified Passam tube used in this study.

This real case is a bit more complicated because there are four instead of two different sections in the tube. But the calculation follows the principle described above. Using a diffusion coefficient of 0.154 cm²/min [4] the result for the theoretical uptake rate is

$UR_{\text{theor}} = 0.727$ cm³/min (20 °C)
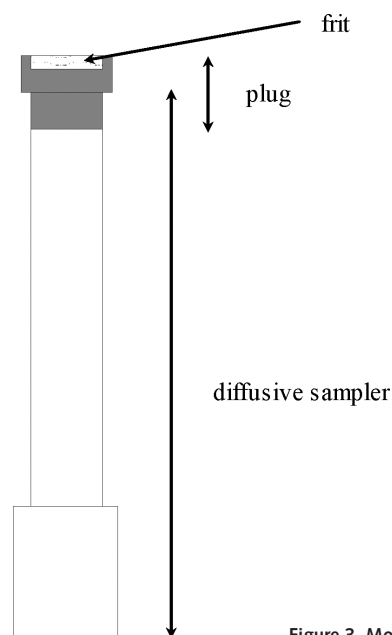
Figure 3. Modified Passam tube (schematic).

Table 1. Results of the equivalence test.

RAW DATA		
Regression	**1.02y – 1.1**	
Regression (*i* = 0)	**1y**	
N	143	n
Outliers	1	n
Outliers	0.7 %	%
Mean CM	53.71	µg/m³
Mean RM	53.65	µg/m³
Number of RM > 0.5 LV	141	n
Number of RM > LV	124	n
REGRESSION RESULTS (RAW)		
slope b	**0.98**	
uncertainty of b	0.02	
intercept a	**1.07**	
uncertainty of a	1.36	
r²	0.91	
slope b (forced trough origin)	**1.00**	significant
uncertainty of b (forced)	0.00607	

EQUIVALENCE TEST (RAW)		
Uncertainty of calibration	1.68	µg/m³
Uncertainty of calibration (forced)	0.24	µg/m³
random term	3.99	µg/m³
additional uncertainty (optional)		µg/m³
bias at LV	0.32	µg/m³
combined uncertainty	4.00	µg/m³
expanded relative uncertainty	**20.0%**	**pass**
ref sampler uncertainty	0.00	µg/m³
limit value	40	µg/m³

The difference to the value found in the experimental evaluation is only 1%. Bearing in mind that for this theoretical estimation various assumptions had to be made this excellent agreement may partly be a fluke. Nevertheless, it underpins clearly the plausibility of the experimental results.

The manufacturer Passam AG also performed a validation for a similar version of their tube equipped with a PE-membrane instead of the glass frit. They calculated an uptake rate of 0.7283 cm³/min and determined experimentally a value of 0.7408 cm³/min [11]. These results are also in excellent agreement with the results of this study.

Orthogonal regression
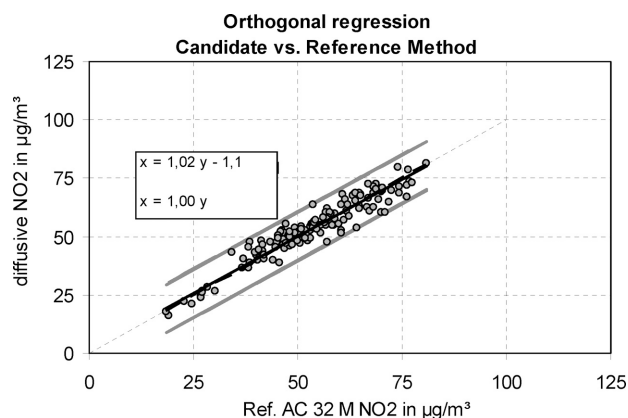**Candidate vs. Reference Method**

Figure 4. Equivalence test based on orthogonal regression.

3.3 Test of equivalence with the reference method EN 14211

The evaluation of potential equivalence of the diffusive sampler method with the chemiluminescence reference method defined in EN 14211 is based on the "Guide to the demonstration of equivalence of ambient air monitoring methods" (GDE) [12] of January 2010. According to section 7.4.3.3.2 of the GDE the average values of the reference method and of the diffusive samplers (averages of the duplicate samples) were entered into a validated spreadsheet provided by the National Institute for Public Health and the Environment (RIVM, Bilthoven, The Netherlands; version 2.6). The spreadsheet option "calibration" was not used because the method was already calibrated by the determination of the uptake rate; one outlier was excluded. The results of the equivalence test are shown in **Table 1** and illustrated in **Figure 4**. The slope of the regression line is 1.02 with an offset of - 1.1 µg/m³. When forcing the regression line through zero the slope of the regression line is 1.00. The expanded uncertainty for a single measurement (exposure over approx. one month) was found to be 20.0%. The data quality objective for indicative measurements of Directive 2008/50/EC (25%) is therefore clearly met. The modified Palmes tubes are suited for air quality measurement in the framework of this Directive. The expanded uncertainty of 20.0% should be a conservative estimation because the uncertainty of the reference method was set to zero for all calculations. The uncertainty is in good agreement with earlier evaluations performed with data from 2004/2005 [5].

It is stressed again that the evaluation described above is based on the assumption that the diffusive method is always operated using duplicate samples. This is the case in all routine measurements in the air quality network in North Rhine-Westphalia.

A comparison of annual means measured in 2009 underlines again the good agreement of the methods (**Figure 5**).

3.4 Measurement uncertainty

According to section 3.3 the expanded uncertainty of single values amounts 20.0% related to the limit value of 40 µg/m³. Assuming that the data have no bias because of the calibration with the reference method, the expanded measurement uncertainty of an annual average could be principally calculated as:

$$U(\mathrm{NO_2})_A = \frac{U(\mathrm{NO_2})_S}{\sqrt{n}} = \frac{U(\mathrm{NO_2})_S}{\sqrt{12}} = \frac{8.00\ \mu g/m^3}{\sqrt{12}} = 2.31\ \mu g/m^3\ (10)$$

$U(\mathrm{NO_2})_A$ Expanded uncertainty (95%) of annual average
$U(\mathrm{NO_2})_S$ Expanded uncertainty (95%) of a single value (one month)
n Number of samples (12 measurements per year)

This estimation implies that no systematic differences exist. For the annual mean calculated for all nine stations this seems to be true: the difference is only 0.2 µg/m³. Systematic influences that may occur at individual stations are not considered in such a simple approach. This is clearly shown by differences between the reference method and the diffusive samplers at single stations which are higher than 2.31 µg/m³ (**Table 2**). An important reason for a bias at individual station may, for example, be due to data captures of the continuous measurements which are significantly lower that 100% whereas data capture of the diffusive tubes is normally 100%.

**Annual Averages 2009
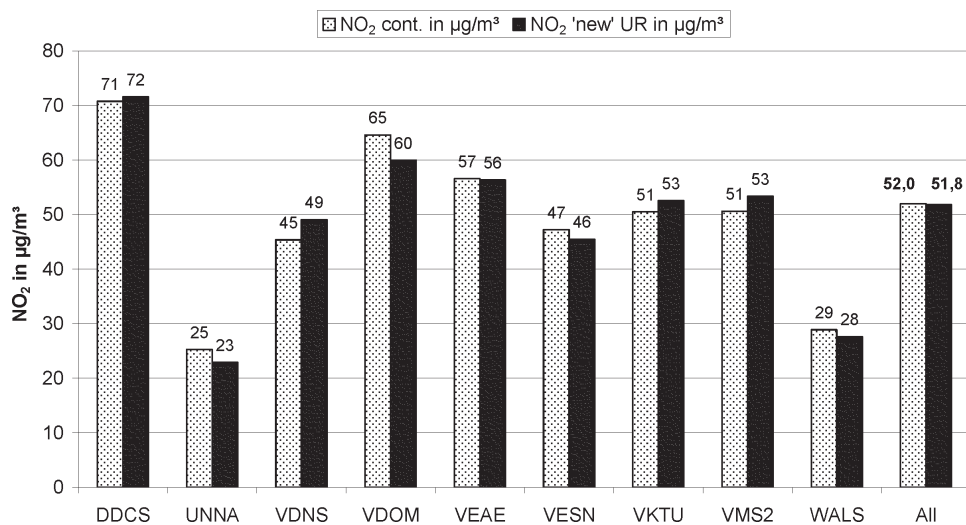Reference Method (AC 32 M) and Diffusive Samplers (UR = 0,734 ml/min)**

Figure 5. Comparison of annual means 2009 (reference method (cont.) and diffusive samplers).

Therefore, the measurement uncertainty of an annual mean at a station is assessed using the squared differences of averages of the reference method and the diffusive samplers:

$$u_V = \sqrt{\frac{\Sigma\left(\bar{y}_{i,diff} - \bar{y}_{i,ref}\right)^2}{n}} \qquad (11)$$

with

u_V standard uncertainty of an annual mean (diffusive) based on differences to the reference value

n number of stations

$\bar{y}_{i,diff}$ annual mean of diffusive sampler at station i

$\bar{y}_{i,ref}$ annual mean of reference method at station i

The results of the uncertainty estimation can be summarised as shown in **Table 3**.
This result documents that annual means of diffusive samplers calculated from 12 monthly averages can also meet the requirements according to Annex I of Directive 2008/50/EC for fixed measurements (15% in the region of the limit value of 40 µg/m³).

3.5 Comparability of laboratories

In order to check the influence of different laboratories applying the Saltzman method [13] for the analysis of the diffusive samplers 113 samples of the year 2009 from randomly selected stations were analysed in parallel by the LANUV laboratory and the lab of Passam AG (Switzerland). The results are illustrated in **Figure 6**.
The regression was calculated using Microsoft Excel's RGP-function which delivers also the following information:

$y = m \cdot x + b$ regression function
m slope
b intercept
$u(m)$ standard uncertainty of the slope
$u(b)$ standard uncertainty of the intercept
R^2 coefficient of determination
$S_{x,y}$ standard error of estimated value for y
F F-value
df degrees of freedom

$ssreg$ sum of squares of regression
$ssresid$ sum of squares of residuals
The results are shown in **Table 4**.
Slope and intercept are not significantly different from one or zero. The residual standard deviation is 3.4 µg/m³ and therefore lower than the combined uncertainty of a single value (4 µg/m³). The expanded uncertainty of the slope is 6% (k = 1.98; 95%).
The standard deviation for parallel measurements was calculated according section 7.4.3 of the GDE:

$$s_V = \sqrt{\frac{\Sigma\left(y_{i;1} - y_{i;2}\right)^2}{2n}}$$

Table 2. Annual mean values for NO₂ at the nine monitoring stations, determined with the reference method and with diffusive samplers.

Station Code	NO$_2$ reference in µg/m³	NO$_2$ diffusive in µg/m³	Difference in µg/m³
DDCS	70.8	71.6	0.79
UNNA	25.3	22.9	-2.37
VDNS	45.4	49.1	3.70
VDOM	64.6	60.0	-4.61
VEAE	56.6	56.4	-0.24
VESN	47.2	45.5	-1.73
VKTU	50.5	52.6	2.05
VMS2	50.6	53.4	2.73
WALS	28.9	27.6	-1.26
All	52.0	51.8	0.20
u_V	2.53		
U(95%)	5.05	12.6 %	

Table 3. Results of the uncertainty estimation for monthly single values and the annual mean.

concentration	Expanded uncertainty (95%)	
	single value (1 month)	Annual mean
40 µg/m³	8.00 µg/m³	5.05 µg/m³
	20.0 %	12.6 %

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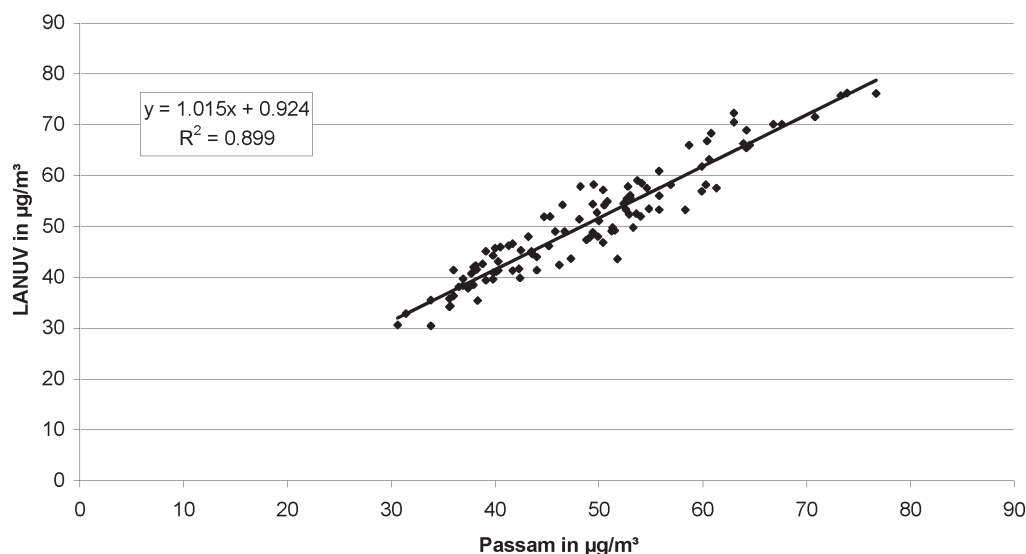## Saltzman Analyses - Comparison of laboratories

Figure 6. Comparison of Saltzman analyses of two laboratories (Passam AG, LANUV; *n* = 113).

$y_{i;1}$ data lab 1 (Passam AG)
$y_{i;2}$ data lab 2 (LANUV)
n number of data pairs

Table 4. Regression analysis of the data of the interlaboratory comparison test.

	m	b	
	1.0145	0.9242	
u(m)	0.0323	1.6093	u(b)
R²	0.8989	3.3820	$S_{x.y}$
F	986.5524	111	df
ssreg	11 284.1796	1 269.6172	ssresid

Table 5. Comparison of two data series – t-Test (95 %).

	LANUV	Passam
Mean value	50.4726	48.8381
Variance	112.0875	97.8833
Observations	113	113
Hypothetical difference of means	0	
Degree of freedom (df)	223	
t-statistic	1.1991	
P (T ≤ t) one-sided	0.1159	
Critical t-value for one-sided t-test	1.6517	
P (T ≤ t) two-sided	0.2318	
Critical t-value for two-sided t-test	1.9707	

Table 6. Comparison of two data series – F-Test (95 %).

	LANUV	Passam
Mean value	50.4726	48.8381
Variance	112.0875	97.8833
Observations	113	113
Degree of freedom (df)	112	112
Test value (F)	1.1451	
P (F ≤ f) one-sided	0.2373	
Critical F-value for one-sided t-test	1.3664	

The standard deviation amounts $s_v = 2.69$ µg/m³ and is similar to the repeatability standard deviation of the Passam AG laboratory (2.38 µg/m³).
With the data from Figure 6 an F-Test and a t-Test were performed on the basis of independent series of measurements as described in VDI 2449 Part 1, section 2.7.2 [14]. No differences of the data series (mean value, variance) could be detected using these tests (**Tables 5** and **6**).

4 Conclusions

For many years, diffusive samplers (modified Palmes tubes) for nitrogen dioxide have been used in the North Rhine-Westphalia air quality network. After a large upgrade of automated measuring systems with monitors representing the reference method EN 14211 a comprehensive re-validation of the diffusive method was performed. The uptake rate of the samplers was evaluated by comparison with the reference method using 286 data pairs (approx. one month exposure) from nine different monitoring stations over three years (2007 to 2009). The determined uptake rate is 0.734 ± 0.004 cm³/min. This value is fully in-line with theoretical estimations of the uptake rate.
Evaluations according to the European "Guide to the demonstration of equivalence of ambient air monitoring methods" reveal an expanded uncertainty of 20.0 % for a single value (monthly average) using a conservative approach. The expanded uncertainty of annual means calculated from 12 monthly averages is found to be 12.6 %. Measurements with diffusive samplers as described in this article can therefore meet the data quality objectives for fixed measurements according to Annex I of the European Directive 2008/50/EC. Data measured with diffusive samplers are equivalent to those of the reference method EN 14211.

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