Laboratory tests of Draeger Polytron 8000 with FL- 6813260 sensor for NH3 measurement

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LIVESTOCK RESEARCH
WAGENINGEN UR

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This research was conducted by Wageningen UR Livestock Research, commissioned and funded by the Ministry of Economic Affairs, within the framework of Policy Support Research theme
'Emission reduction' (project number BO-20-0004-049 /BO-20-004-093)

Wageningen UR Livestock Research
Wageningen, August 2016

Livestock Research Report 972

Melse, Roland W.; Johan P.M. Ploegaert; Nico W. M. Ogink, 2016. *Laboratory tests of Draeger Polytron 8000 with FL–6813260 sensor for NH_3 measurement.* Wageningen UR (University & Research centre) Livestock Research, Livestock Research Report 972.
http://dx.doi.org/10.18174/388326

Samenvatting NL: Twee Draeger Polytron 8000 instrumenten met een FL–6813260 NH_3 sensor zijn getest in kortdurende laboratoriumexperimenten in het bereik van 0 to 11.0 ppm. De meetonzekerheid bleek lager te zijn dan door de fabrikant werd opgegeven. In het gebied van 1 - 11 ppm is de nauwkeurigheid van de sensor voldoende hoog om deze toe te passen ten behoeve van emissiemonitoring. In het gebied van 0 - 1 ppm is echter een grotere nauwkeurigheid nodig om de sensor te kunnen toepassen in natuurlijk geventileerde melkveestallen. De conclusie is dat de sensor een veelbelovende apparaat is voor ammoniakmetingen in en rond stallen, en geschikt is om nader te evalueren onder praktijkomstandigheden. Om de werking van de sensor en de stabiliteit van de kalibratielijnen vast te stellen op de lange termijn en onder praktijkomstandigheden, wordt aanbevolen om de Draeger sensor in een veldstudie nader te testen.

Summary UK: Two Draeger Polytron 8000 instruments with FL–6813260 sensor for NH_3 measurement were tested in short-term laboratory experiments in the range from 0 to 11.0 ppm. The measurement uncertainty found in the calibration tests was lower than specified by the manufacturer. In the 1-11 ppm range the measurement accuracy is sufficient to meet requirements for emission monitoring. However higher accuracy levels are needed in the 0-1 ppm range to be of interest for application in naturally ventilated cattle barns. It is concluded that the sensor is a promising device for ammonia measurements in and around animal houses, and is suitable for further evaluation under practical conditions. In order to monitor the sensor performance and the stability of calibration lines on long term under barn conditions, it is recommended to test the Draeger sensor in a field study.

Livestock Research Report 972

Table of contents

1 Introduction

In naturally ventilated cattle houses, ammonia (NH_3) concentrations in a range of 0.1 - 3 ppm[1] can be expected under summer conditions and about 1 - 6 ppm under winter conditions. In mechanically ventilated pig houses, concentrations can be tenfold. At many pig houses air scrubbers are installed that reduce the ammonia concentrations in the air down to a range of 0.1 - 3 ppm, which is similar to the concentrations that are found in dairy cattle houses in summer.
In the Netherlands a regulatory system is in place which applies an NH_3 emission factor to each animal house based on prescribed design and management practices. This means that the actual emissions from the farms are not monitored, although a large variation in emission levels may exist between farms. A similar system with design-based emission factors is used for air scrubbers and in addition several secondary parameters are measured (pH and EC of trickling water, electricity use, pressure drop over packing, fresh water use and discharge water amount). These parameters are stored electronically using a so-called obligatory "electronic monitoring system". The values and development of these parameters are used for verifying proper scrubber operation and as a consequence, are used for assessing sufficient ammonia removal performance of the scrubber.
Currently no robust and reliable sensor is available for long-term continuous monitoring of NH_3 concentrations in animal houses or air scrubbers at low costs. Especially low concentrations (< 1 ppm) and high humidity (outlet scrubber air) make it difficult to operate sensors for a long period (sensor deteriorates fast and frequent zero-calibration is necessary).
However, a suitable sensor that could continuously measure NH_3 would enable farmers to real-time monitor the ammonia concentrations in the animal house (e.g. for welfare reasons), the actual emissions from the animal house and the performance of the air scrubber. For monitoring the NH_3 emission from an animal house, the NH_3 concentration measurement could be combined with a CO_2 concentration measurement, the latter enabling an estimation of the air flow rate by means of the CO_2-balance method. For monitoring the performance of an air scrubber the NH_3 concentration of both inlet and outlet air could be measured.
The German company Draeger has developed a new NH_3 sensor that is suitable for a range of 0.5 - 100 ppm according to the specifications. Draeger states that this sensor is expected to be suitable for long-term use without the need of regular recalibration (low zero-drift) or other maintenance.
The aim of this study is to carry out a short-term laboratory test of this sensor in order to find out how this sensors performs with regard to detection limit and measurement accuracy, especially at the lower concentrations (≤ 11 ppm). Also the aim is to test whether or not the measurement is influenced by the relative humidity of the air.
This study was funded by the Dutch Ministry of Economic Affairs. The sensors were provided by Draeger at no cost.

[1] 1 ppm NH_3 = 0.70 mg/m^3 at 25°C and 1 atm.

2 Material and Methods

2.1 Sensors and data processing

In all experiments two NH_3 sensors were used that were connected parallel, generating in duplo measurements.
Sensor name and type: Draeger Polytron 8000 with FL–6813260 sensor for NH_3
Main specifications according to manufacturer:

- Working principle: electrochemical diffusion sensor;
- Detection limit: 1 ppm;
- Measurement range limit (max): 100 ppm;
- Calibration: the sensor is factory-calibrated with NH_3 and zero gas (2-point calibration);
- Measurement uncertainty: ≤ ±5% of measured value with a minimum of ≤ ±1.5 ppm (the greater value applies);
- Operation temperature and relative humidity: -40/65°C and 15/95% (although the measurement principle does not require water in the air, Draeger recommends to have an humidity of > 10% when the sensor is purged constantly, i.e. > 5 days);
- Air pressure: ambient (1 bar) ± 1%;
- Cross references: in Table 1 the known cross-references are given as supplied by the manufacturer;
- Output signal: 4...20 mA.

Table 1
Known cross-references for Draeger NH_3 sensor FL–6813260.

Test gas	Concentration (ppm)	NH_3 sensor reading (ppm)
CO	100	< 0.5
H_2S	20	< 0.8
NO_2	10	< 0.7
NO	20	< 0.5
CO_2	16,000	< 0.5
SO_2	20	-12
H_2	1,000	< 0.5
Cl_2	10	-6
C_2H_4	1,000	< 0.5
HCN	50	< 0.5
HCl	5	< 0.5
O_3	0.5	< 0.5
C_2H_5OH	250	< 0.5
H_2O_2	10	< 0.5
Limonene[a]	saturated	< 0.5
Peracetic acid	saturated	< 0.5
N_2O	10,000	< 0.5

[a] 1-Methyl-4-(1-methylethenyl)-cyclohexene; [b] CH_3CO_3H.

2.2 Experimental setup

Two NH_3 gas cylinders were used, containing 11.0 ppm and 101 ppm in N_2 (Scott Specialty Gases, see certificates in Appendix A and B). The gas from the cylinder was diluted with compressor air using two adjustable gas diluters and led to a measurement chamber were the two NH_3 sensors are placed. In Figure 1 a schematic of the setup is given; in Figure 2 both NH_3 sensors are shown.
Gas diluter #1 is a Signal Model 821 Gas Divider, which has an adjustable dilution ratio up to 1 : 10. The dilution ratio is set with percentages ranging from 10% (i.e. 10% source gas is mixed with 90% of compressed air, so 10 x dilution) to 100% (i.e. 100% source gas without compressed air, no dilution). If necessary, a second gas diluter (#2) was connected in series. Gas diluter #2 is an EPM Diluting Stack Sampler, diluting unit 797-430, adjustable dilution ratio) which was operated at a dilution ratio of 1 : 18.65 (at a pressure of 6.0 bar). In Appendix C and D the calibration reports from both gas diluters are given In one experiment a humidifier was connected in order to increase the humidity of the air prior to reaching the NH_3 sensors. The humidifier consisted of a bottle with at the bottom a small amount of water (net air volume of bottle, incl. extra tubing: 380 ml). The bottle is placed in a thermostat bath. Through the lid of the bottle an inlet and outlet tube are inserted; the inlet tube is close to the water level but does not touch it, the outlet tube is close to the top of the bottle. By increasing the temperature of the bath, the humidity of the air increases. Before reaching the sensors, the humidity and temperature of the air are measured (Vaisala, Finland; HMP 110). Then the air reaches a chamber where the NH_3 concentrations are measured with the two Draeger Polytron 8000 FL–6813260 sensors. With a precision resistor of 50 Ω (Neohm UPW-50, ± 0.1%, 3x10-6 Ω/°C) the 4 - 20 mA output signal of the sensors was converted to a 200 - 1000 mV signal that was recorded every 10 seconds with a data logger (Campbell Scientific Inc., Logan UT, USA; type CR1000). The resolution of the data logger is 0.1 mV (i.e. 0.0125 ppm) for values between 200 and 800 mV (i.e. 0 - 75 ppm) and 1 mV (i.e. 0.125 ppm) for values above 800 mV (i.e. > 75 ppm). Eventually the measurement by the Draeger sensors are converted to ppm's by assuming 4 mA is equal to 0 ppm and 20 mA is equal to 100 ppm, in accordance with the specifications of the sensor.
All tubing was made of FEP in order to prevent absorption of ammonia. The experiments were carried out at room temperature (20-23°C) and at atmospheric pressure (1015-1022 hPa); the gas temperature at the sensors was slightly higher (23-25°C). During the experiments no condensation was observed in the tubing or in the measurement chamber.
Prior to the experiments the used equipment was checked and calibrated:
- the data logger values were checked and calibrated with a calibrated voltmeter (Fluke, type 87III);
- the value of the precision resistors were checked with a calibrated voltmeter (Fluke, type 87III);
- the gas dilution ratio of both gas diluters was based on available certificates and checked using a chemiluminescent NO_x analyser (Monitor Labs / API, model 200 A) and a cylinder of NO gas (Scott Specialty Gases, 40 ppm).

2.3 Experiment 1: Detection limit and accuracy at low concentrations

In this experiment a range of NH_3 concentrations between 0 en 11 ppm was supplied to the NH_3 sensors, using different gas cylinders and diluter settings. The humidification unit was switched off. Table 2 explains how the different NH_3 concentrations were generated. Also a zero ppm NH_3 test was done by supplying pure compressed air to the sensors. Each concentration was supplied for about 15 minutes in which period a stable signal could be established. During these experiments the relative humidity was 3-5% and the temperature 23-25°C.
The concentration level that was supplied at the sensors was calculated based on the gas diluter settings and the source gas concentration.

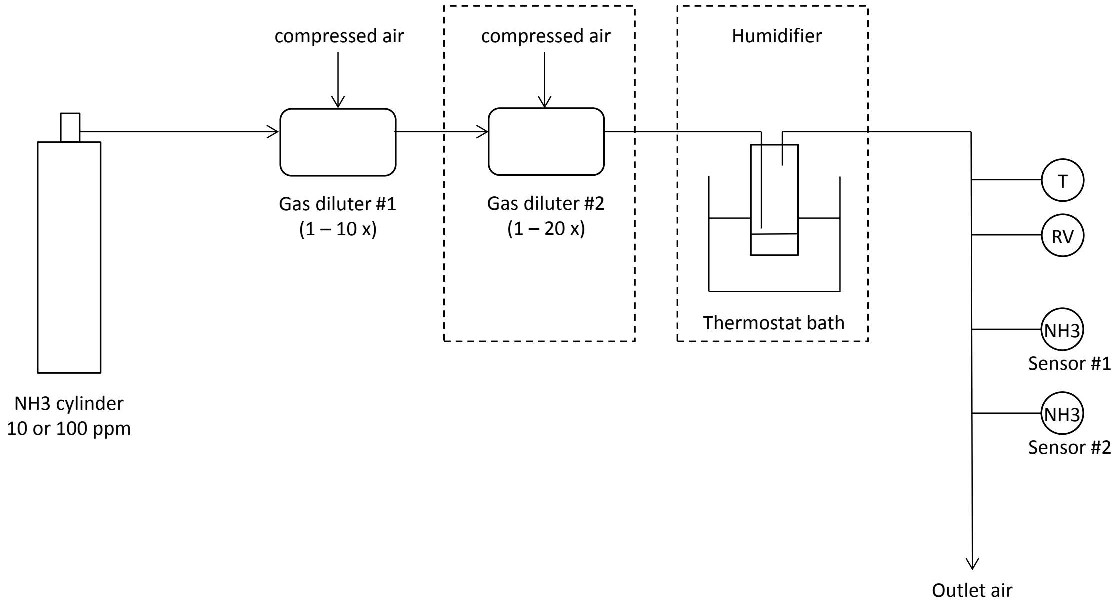

Figure 1 *Schematic of laboratory setup for testing the NH_3 sensors.*

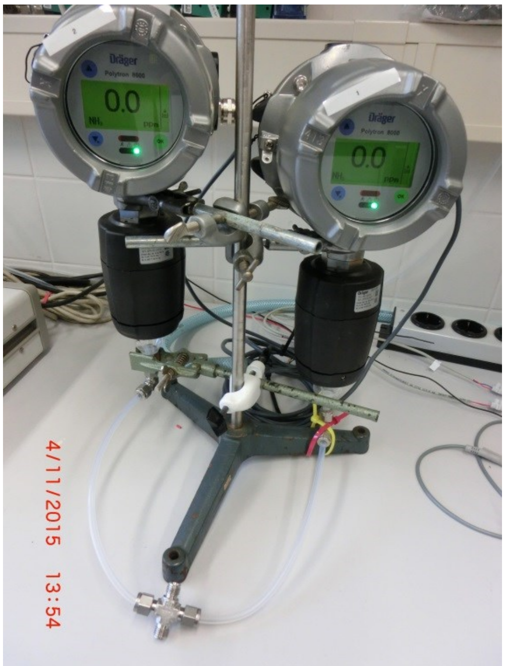

Figure 2 *Picture of the two NH_3 sensors.*

Table 2

Test concentrations of NH_3 - low concentration range (0 - 11 ppm); n.a. = not applicable.

Source NH_3 gas (ppm)	Gas diluter #1 (%) SIGNAL	Gas diluter #1 (times diluted) SIGNAL	Gas diluter #2 (times diluted) EPM 6.0 bar	Supplied NH_3 at sensors (ppm)
0	n.a.	n.a.	n.a.	0
11.0	10	9.95	18.65	0.06
11.0	20	5.02	18.65	0.12
11.0	40	2.52	18.65	0.23
11.0	80	1.25	18.65	0.47
11.0	100	1.00	18.65	0.59
11.0	10	9.95	n.a.	1.11
11.0	20	5.02	n.a.	2.19
11.0	30	3.35	n.a.	3.28
11.0	40	2.52	n.a.	4.37
11.0	50	2.00	n.a.	5.49
11.0	80	1.25	n.a.	8.79
11.0	100	1.00	n.a.	11.0

2.4 Experiment 2: Influence of relative humidity

In this experiment a selection of NH_3 concentrations was fed to the NH_3 sensors, using the humidification unit to establish different humidity levels of the gas. Gas diluter #2 was switched off. In Table 3 the testing conditions are further explained. Also a zero ppm NH_3 test was done by supplying pure compressed air to the sensors. Each concentration was supplied for about 15 minutes in which period a stable signal could be established. During these experiments the temperature was 23-25°C.

Table 3

Test concentrations of NH_3 at low relative humidity (3-5%), medium (40-50%), and high relative humidity (70-80%); n.a. = not applicable.

Source NH_3 gas (ppm)	Gas diluter #1 (%) SIGNAL	Gas diluter #1 (times diluted) SIGNAL	Supplied NH_3 at sensors (ppm)
0	n.a.	n.a.	0
11.0	10	9.95	1.11
11.0	50	2.00	5.49
101	10	9.95	10.2

3 Results and discussion

3.1 Experiment 1: Detection limit and accuracy at low concentrations

In Table 4 the results of experiment 1 are shown, after stabilization of the output signal of the sensors for at least 5 minutes. The "supplied NH_3 concentration" is the concentration as calculated from the source gas and the dilution equipment settings. After the concentration was increased, after about 3 minutes a stable output signal was achieved. Within the experimental setup that was chosen it is not possible to distinguish between the time that is needed to reach the desired concentration in the air present at the sensors (as it is no perfectly mixed system), or the time that is needed by the sensors to respond to a step increase or decrease of ammonia concentration of the air supplied to the sensor.

Table 4
Results of NH_3 measurements with electrochemical diffusion sensors - low concentration range (temperature: 23-25°C; relative humidity: 3-5%); n.d. = not detected.

Supplied NH_3 concentration (ppm)	Measured NH_3 concentration (ppm)	
	Sensor #1	Sensor #2
0	-0.19	-0.18
0.06	n.d.	n.d.
0.12	n.d.	n.d.
0.23	-0.025	0.068
0.47	0.31	0.41
0.59	0.40	0.49
1.11	1.15	1.24
2.19	2.25	2.35
3.28	3.43	3.53
4.37	4.69	4.76
5.49	5.78	6.04
8.79	9.14	9.40
11.0	11.25	11.68

For the tested concentrations of 0.06 and 0.12 ppm, no increase of the mA output signal of the sensors was detected, therefore these values are reported as "not detected". As can be seen for the 0 ppm value (and for the 0.23 ppm value for sensor #1) slightly negative measured ppm values were found, i.e. the output signal was slightly lower than 4 mA. From Table 4 it follows that both sensors overestimate the supplied NH_3 at values between 1.11 and 11.0 ppm, and underestimate at concentrations of 0.59 ppm and lower.

In Figure 3 the results of Table 4 are plotted, together with a linear regression line for each sensor. This regression line can be considered as the calibration line to be used for each sensor. Furthermore, the line $Y = X$ is shown. The regression lines can be formulated as (R2 = coefficient of determination; RSD = Residual Standard Deviation):

Sensor #1:
Measured [NH_3] (ppm) = Supplied [NH_3] (ppm) x 1.0506 - 0.1143
[R2 = 0.9989; RSD = 0.137]

Sensor #2:
Measured [NH_3] (ppm) = Supplied [NH_3] (ppm) x 1.0803 - 0.0691
[R2 = 0.9993; RSD = 0.113]

The concentration that can be calculated using these calibration lines and the measured values, is called the predicted concentration.

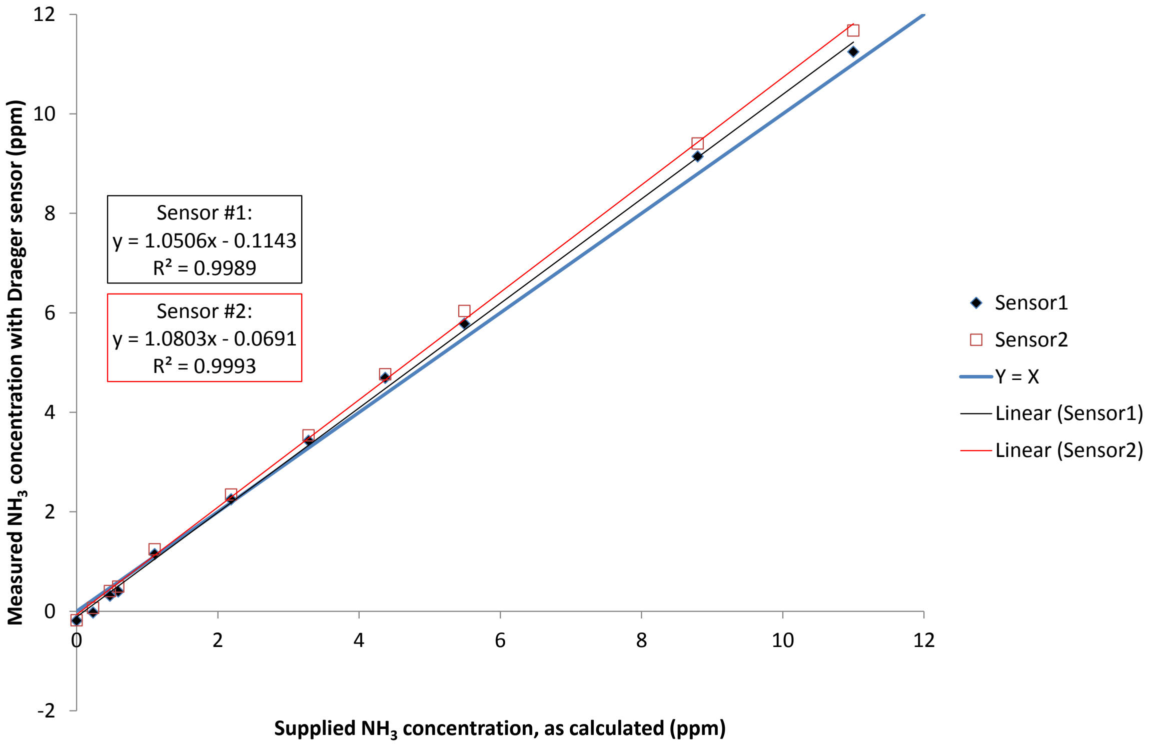

Figure 3 *Measured NH_3 concentration versus supplied NH_3 concentration for both Draeger sensors (RV = 3-5%).*

When the measurement signal (in mA) is used as input variable and the supplied NH_3 concentration as output, the following equations can be formulated:

Sensor #1:
Supplied [NH_3] (ppm) = Measurement signal (mA) x 5.9425 - 23.658
[R2 = 0.9989; RSD = 0.13]

Sensor #2:
Supplied [NH_3] (ppm) = Measurement signal (mA) x 5.7813 - 23.059
[R2 = 0.9991; RSD = 0.10]

In Figure 4 the residuals are shown, i.e. the deviations between supplied and the predicted concentrations. Figure 4 clearly shows that residuals are not evenly distributed over the whole concentration range. In the range < 1 ppm the predicted concentrations are lower than the supplied concentrations (positive residual values) and in the range 1 - 6 ppm the predicted concentrations are higher than the supplied concentrations (negative residual values). The highest measurements then show positive residual values) again. The residuals show that the sensors do no respond rectilinear in the offered concentration range. For this reason the calibration lines and residual values were calculated when the concentration range is split into a 0 - 1 ppm and a 1 - 11 ppm range; this is shown in Figure 5 and 6 for Sensor #1 and #2, respectively.

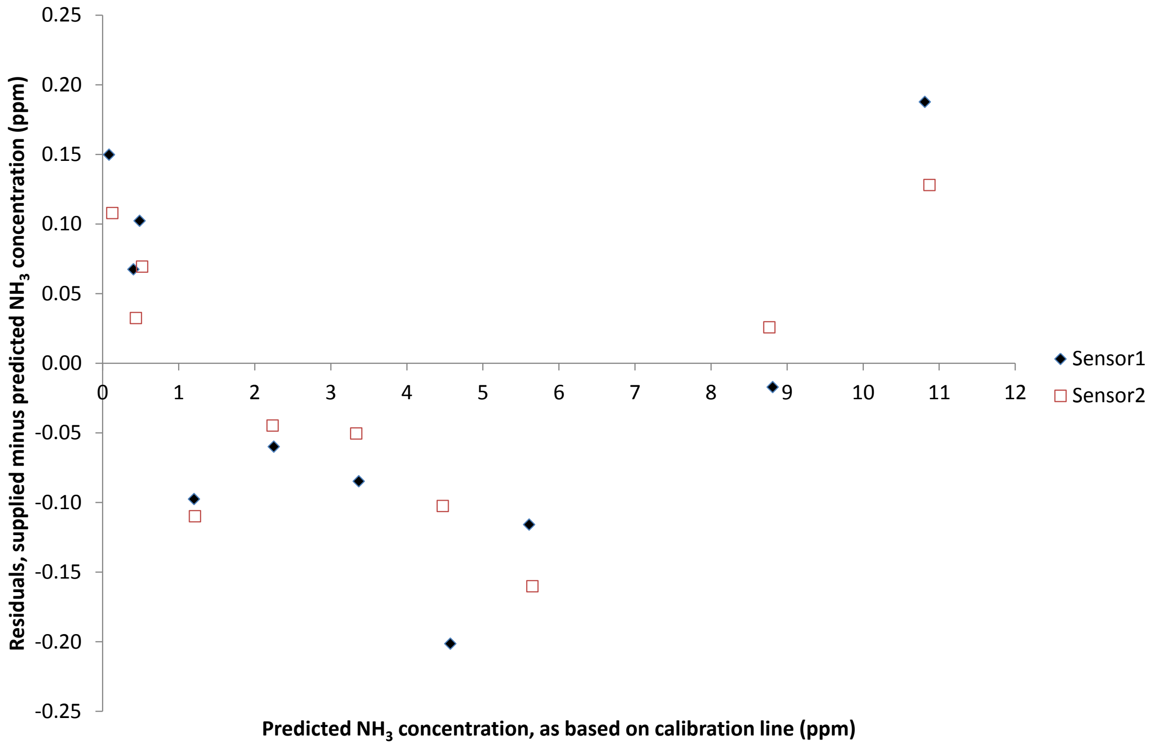

Figure 4 *Residual values (i.e. supplied minus predicted NH_3 concentration) for both Draeger sensors (RV = 3-5%).*

The regression lines shown in Figure 5a and 6a can be formulated as follows:

<u>0 - 1 ppm, Sensor #1:</u>
Measured [NH_3] (ppm) = Supplied [NH_3] (ppm) x 1.2336 - 0.2636
[R2 = 0.9858; RSD = 0.071]

<u>0 - 1 ppm, Sensor #2:</u>
Measured [NH_3] (ppm) = Supplied [NH_3] (ppm) x 1.2958 - 0.2168
[R2 = 0.9954; RSD = 0.042]

<u>1 - 11 ppm, Sensor #1:</u>
Measured [NH_3] (ppm) = Supplied [NH_3] (ppm) x 1.0247 + 0.0781
[R2 = 0.9994; RSD = 0.096]

<u>1 - 11 ppm, Sensor #2:</u>
Measured [NH_3] (ppm) = Supplied [NH_3] (ppm) x 1.0585 + 0.0921
[R2 = 0.9997; RSD = 0.076]

In this way for measurements in the range of 0 - 1 ppm the first regression line can be used for calibration, while in the range of 1 - 11 ppm the second regression line can be used.

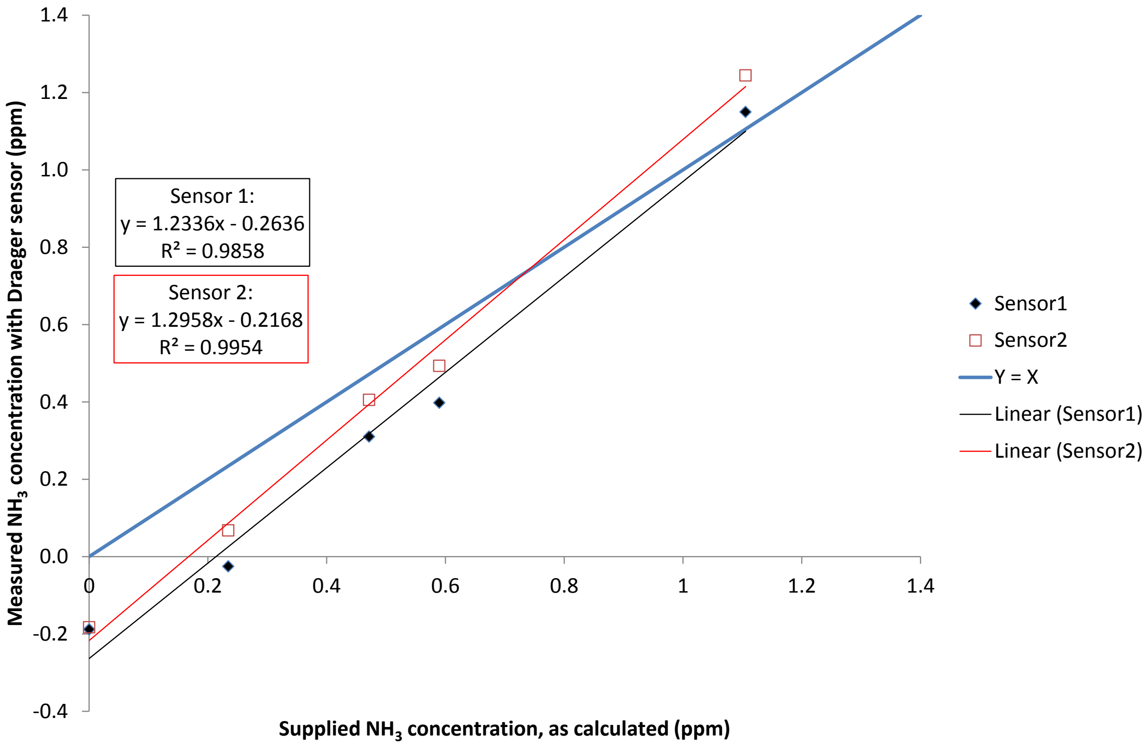

Figure 5a Measured NH_3 concentration versus supplied NH_3 concentration for both Draeger sensors, range of 0 - 1 ppm (RV = 3-5%).

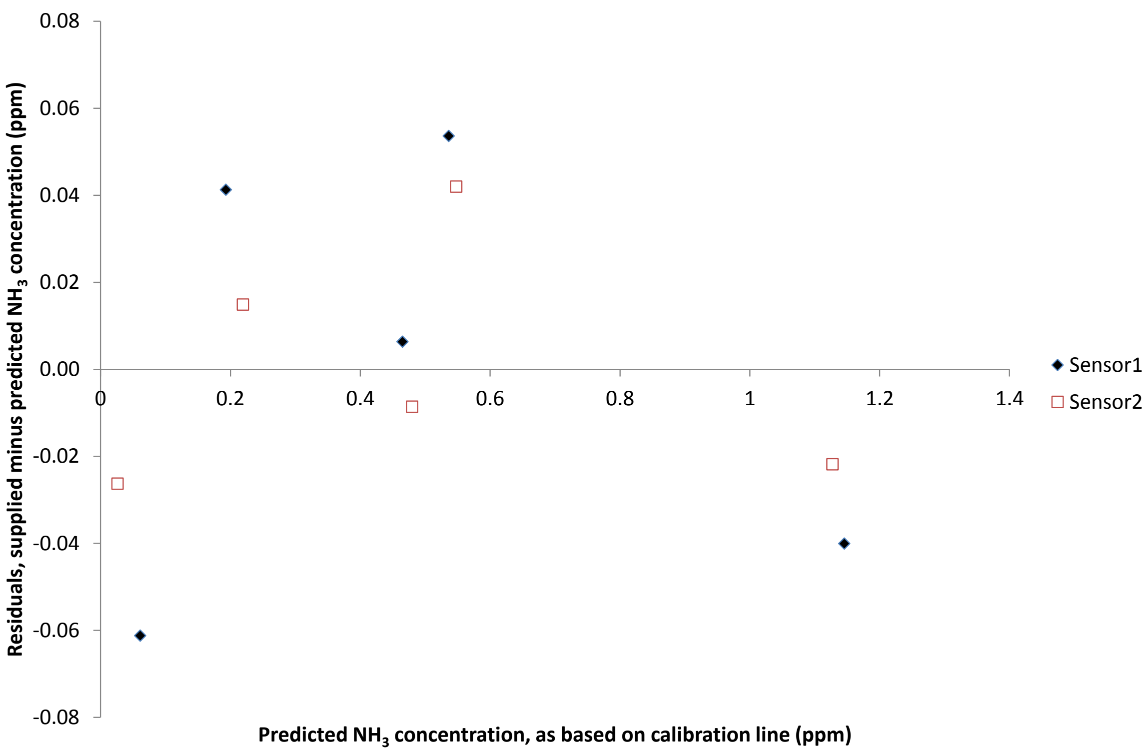

Figure 5b *Residual values (i.e. supplied minus predicted NH_3 concentration) for both Draeger sensors, range of 0 - 1 ppm (RV = 3-5%).*

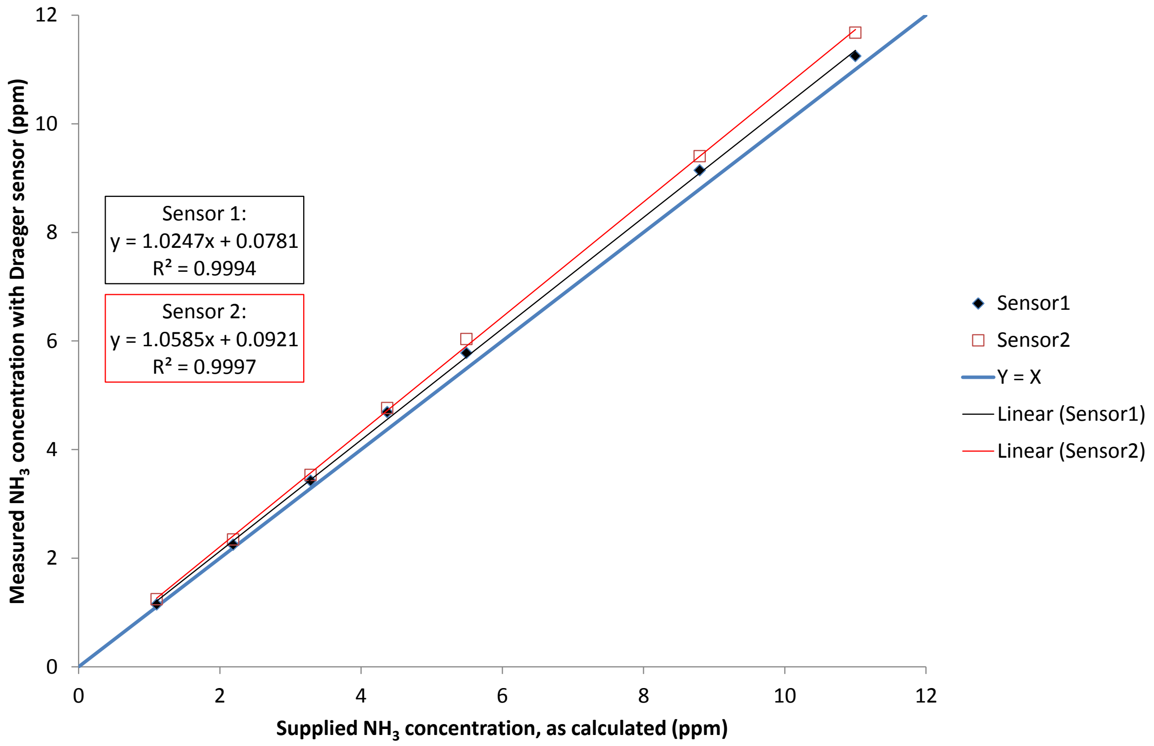

Figure 6a *Measured NH_3 concentration versus supplied NH_3 concentration for both Draeger sensors, range of 1 - 11 ppm (RV = 3-5%).*

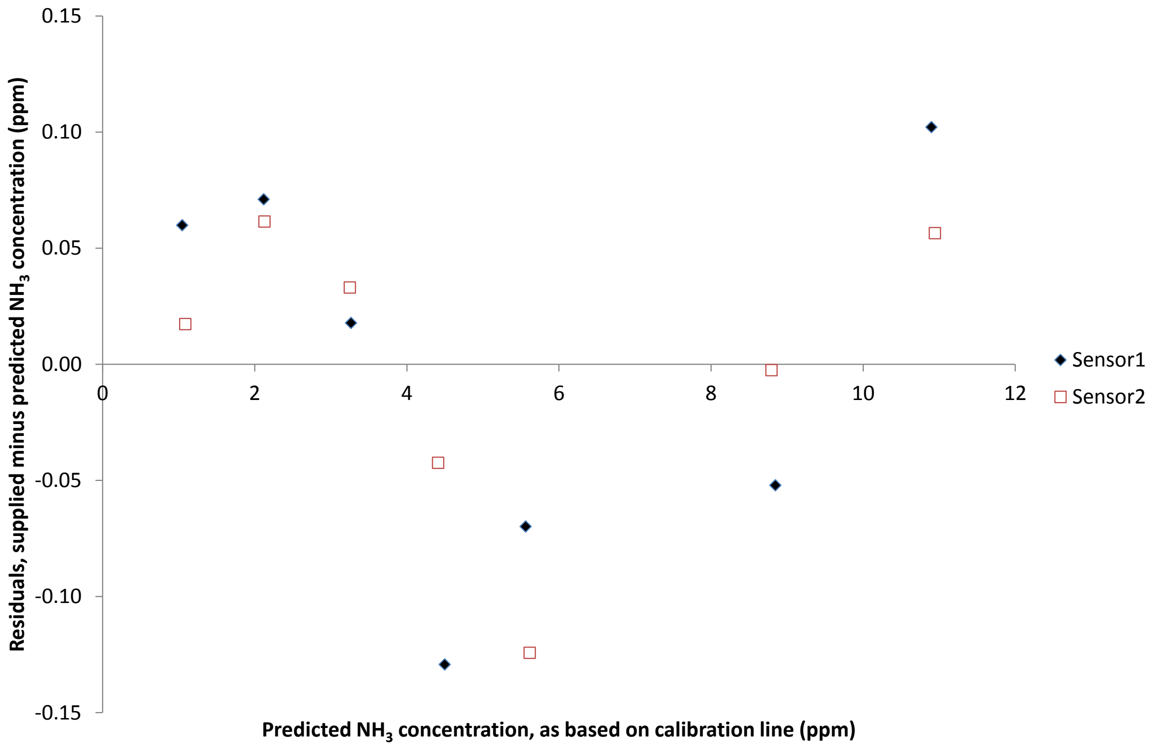

Figure 6b *Residual values (i.e. measured NH_3 concentration minus predicted NH_3 concentration) for both Draeger sensors, range of 1 - 11 ppm (RV = 3-5%).*

Comparison of the residual values in Figure 4 (whole range) with Figure 5b (0 - 1 ppm) and Figure 6b (1 - 11 ppm) shows that the residual values decrease when a different calibration curve is used for the low and the high range concentrations. This means that more accurate measurements can be done.

3.2 Experiment 2: Influence of relative humidity

In Table 5 the results of experiment 2 are shown, after stabilization of the output signal of the sensors.

Table 5
Results of NH_3 measurements with electrochemical diffusion sensors at different humidity levels (temperature: 23-25°C); n.a. = not applicable.

Relative humidity (%)	Supplied NH_3 concentration (ppm)	Measured NH_3 concentration (ppm)	
		Sensor #1	Sensor #2
3-5 (low)	0	-0.10	-0.18
	1.11	1.15	1.24
	5.49	5.78	6.04
	10.2	10.6	10.9
40-50 (medium)	0	-0.103	-0.020
	1.11	1.15	1.24
	5.49	5.95	6.12
	10.2	10.7	11.1
70-80 (high)	0	0.045	0.068
	1.11	1.41	1.49
	5.49	5.98	6.20
	10.2	11.1	11.7

In Figure 7 the average measurement values of the sensors are plotted against the supplied NH_3 concentration for all RV ranges, together with a linear regression line. The regression line for low RV (3-5%) is the calibration line that was presented already in section 3.1. Figure 7 clearly shows that the sensors show higher readings at higher humidity levels, as the lines for medium RV (black) and high RV (red) are above the line for low RV (blue).

Furthermore, in this experiment it took more time for the sensors to stabilize than in Experiment 1, in some cases up to 25 minutes; in Experiment 1 the time needed for stabilization was only about 3 minutes. This delay is caused by the fact that by connecting the bottle for humidification, the gas volume of the measurement system is largely increased. As a result, the time that is needed to reach the desired concentration in the air present at the sensors increases (as it is no perfectly mixed system), thus ending up in a slower responses to concentration changes.

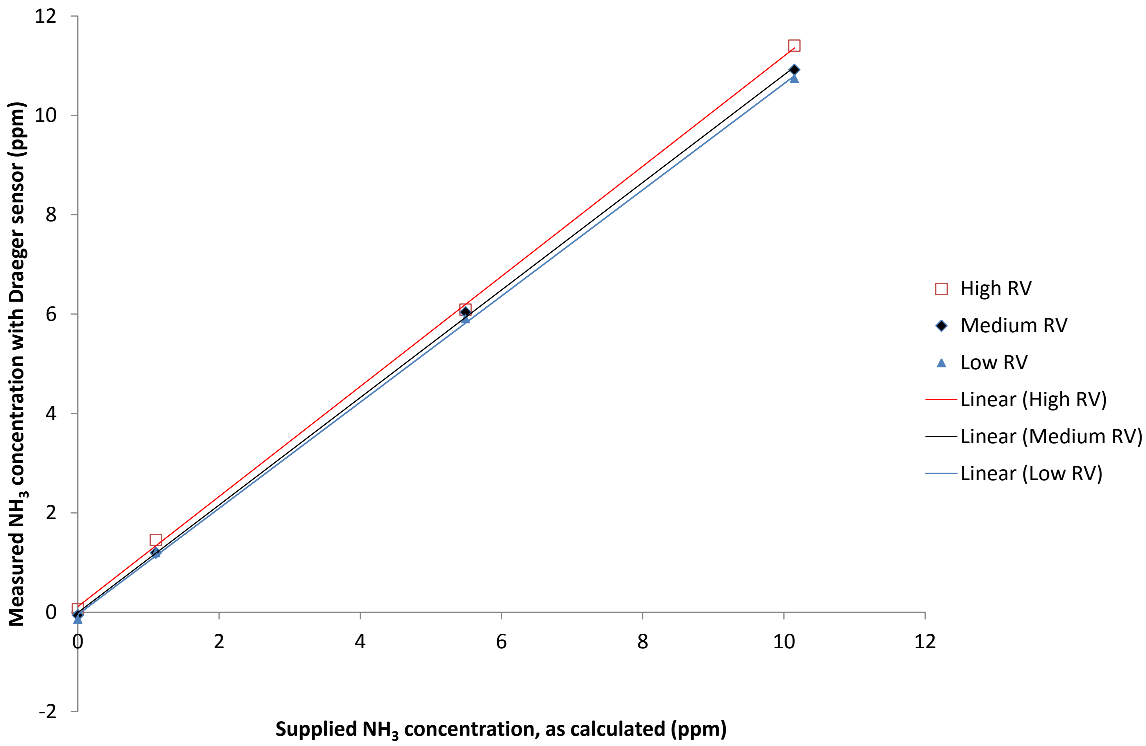

Figure 7 *Measured NH_3 concentration versus supplied NH_3 concentration (average of both Draeger sensors) at different humidity levels.*

3.3 Comparability of sensors

To test the comparability of the results the two different NH_3 sensors were exposed simultaneously to the same polluted air, and the response of the devices to the provided concentration was registered, as described. This test was repeated at different ammonia concentration levels and different relative humidity levels. Then, the dataset with the paired results from the devices was analysed for outliers by using the box plot method with three times the interquartile range (IQR) as measure for extreme outliers:

IQR = Q3-Q1
Q1: lower (first) quartile (25%)
Q2: median (second quartile)
Q3: upper (third) quartile (75%)
Outlier if: value > Q3+3*IQR OR value < Q1-3*IQR

This test shows that devices are giving not comparable results at the low concentration levels, i.e. measured concentrations ≤ 0.5 ppm (supplied concentration: ≤ 0.59 ppm), as all results in this range were found to be outliers. For measured concentrations at and above 1.2 ppm (supplied concentration ≥ 1.11 ppm), no outliers were found. Figure 8a also shows a (small) systematic difference (4%) between both devices. The observed difference between devices is independent of the provided NH_3 concentration level (Figure 8b).

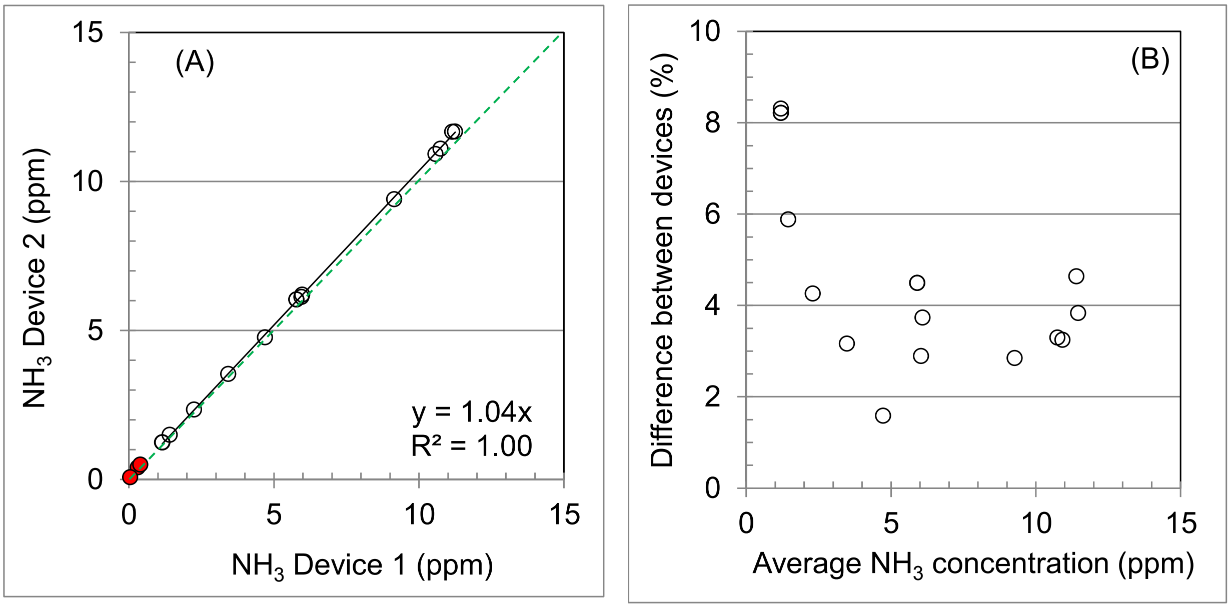

Figure 8 *Comparability of results between devices. (A) Absolute measured values; red dots are the points considered as outliers in the box plot method; (B) Difference between devices vs. average NH_3 concentration.*

4 Conclusions and recommendations

Two Draeger Polytron 8000 instruments with FL–6813260 sensor for NH_3 measurement were tested in short-term laboratory experiments in the range from 0 to 11.0 ppm. The results showed that the response of the sensor was not fully rectilinear in this range, and that calibration with a linear model is best applied separately at the 0 – 1 ppm range and the 1-11 ppm range.

The mean residual standard deviation of the 0-1 ppm calibration line amounted 0.056 ppm. Estimating an uncertainty interval, as an approximation, on two times the standard deviation this would result in an uncertainty interval of ± 0.11 ppm. Expressed as a relative error of the median value of 0.5 ppm in this range, the relative error here would amount 23%. At values in the 1-11 ppm range the residual standard deviation amounted 0.086 ppm, resulting in an uncertainty interval of 0.17 ppm, and a relative error at the median value of 6 ppm of 2.8%. In both intervals we assume that no systematic errors are present because bias is corrected on basis of the regression lines. Our test setup was not suited to evaluate drift from the calibration lines, hence estimated uncertainties represent a best case scenario.
Nevertheless the results give the impression that in the 1-11 ppm range the accuracy is better than expected from the specifications of the manufacturer (Specified measurement uncertainty: ≤ ±5% of measured value with a minimum of ≤ ±1.5 ppm, where the greater value applies). In the 0-1 ppm range the measurement uncertainty amounted ± 0.11 ppm, being considerably lower than the specified minimum of ≤ ±1.5 ppm.
Also, the influence of humidity was tested. The measured NH_3 concentration slightly increased at higher humidity levels.

It is concluded from the laboratory test that this Draeger sensor is a promising device for ammonia measurements in and around animal houses, and is suitable for further evaluation under practical conditions. The measurement uncertainty found in the calibration tests was lower than specified by the manufacturer. In the 1-11 ppm range the measurement accuracy is sufficient to meet requirements for emission monitoring. However higher accuracy levels are needed in the 0-1 ppm range, which range is especially of interest in naturally ventilated cattle barns. For application in practice it is important to monitor the sensor performance and the stability of calibration lines on long term under barn conditions and also its performance at higher concentration levels.

Therefore it is recommended to test the Draeger sensor in a field study. A field study could include:
- Measurements inside a natural ventilated dairy house, combined with CO_2 measurements for estimation of the ventilation rate;
- Measurements of the ammonia concentration levels inside a pig or poultry house;
- Measurements of the ammonia removal efficiency of a scrubber operated at a pig house.

Appendix A - Certificate of 11.0 ppm NH_3 gas cylinder

Scott specialty gases

CUSTOM CLASS

Takkebijsters 48 4817 BL, Breda, The Netherlands Phone: +31(0)76-5711 828 Fax: +31(0)76-5713 267

CERTIFICATE OF ACCURACY: Custom Class Calibration Standard

Product Information
Project No.: 20-97567-002
Item No.: 20020000150Z50L
P.O. No.: WUR728325

Cylinder Number: 5902857
Cylinder Size: 50L
Certification Date: 05May2014
Expiration Date: 04May2015

Customer
WAGENINGEN UR,ANIMAL SCIENCES GROUP
T.A.V. JOHAN PLOEGAERT
VEEHOUDERIJ BV
VIJFDE POLDER 1
6708 WC WAGENINGEN
NEDERLAND

CERTIFIED CONCENTRATION

Component Name	Concentration (Moles)		Accuracy (+/-%)
AMMONIA	11,0	PPM	3
NITROGEN		BALANCE	

TRACEABILITY

Description	Traceability Type	Traceable To
ANALYTICAL TRACEABILITY	GAS STANDARDS	

APPROVED BY: R.J. HOFMEESTER

DATE: 14-05-2014

SUPERVISOR: C. OOMEN

Page 1 of 2

An Air Liquide Group Company

SPECIFICATIONS

Component Name	Requested Concentration (Moles)	Gravimetric Concentration (Moles)	Analyzed Concentration (Moles)	Blend Tolerance Result (+/- %)	Blend Process Accuracy Result (+/- %)	Analytical Accuracy Result (+/- %)	Interlocking Result (+/- %)
AMMONIA	10, PPM	N/A	11,0 PPM	10,0	N/A	3,00	N/A
NITROGEN							

TRACEABILITY

Analytical Traceability

Type/SRM No.	Expiration Date	Cylinder Number	Concentration	Component
SRS TSTD	05May2014		0,000	AMMONIA

PHYSICAL PROPERTIES

Cylinder Size: 50L

Pressure: 150 BAR

Expiration Date: 04May2015

Valve Connection: DIN-5

Min. Cyl. Pressure: 5 BAR

Min. Temperature: -10 'C

SPECIAL HANDLING INSTRUCTIONS

Do not use or store cylinder at or below the stated dew point temperature. Possible condensation of heavier components could result. In the event the cylinder has been exposed to temperatures at or below the dew point, place cylinder in heated area for 24 hours and then roll cylinder for 15 minutes to re-mix.

Use of calibration standards at or below dew point temperature may result in calibration error.

Page 2 of 2

Appendix B - Certificate of 101 ppm NH_3 gas cylinder

Scott specialty gases

CUSTOM CLASS

Takkebijsters 48 4817 BL, Breda, The Netherlands Phone: +31(0)76-5711 828 Fax: +31(0)76-5713 267

CERTIFICATE OF ACCURACY: Custom Class Calibration Standard

Product Information
Project No.: 20-01957-002
Item No.: 20020000150Z50L
P.O. No.: WUR837436

Cylinder Number: 5902919
Cylinder Size: 50L
Certification Date: 30Apr2015
Expiration Date: 29Apr2018

Customer
WAGENINGEN UR,ANIMAL SCIENCES GROUP
ATTN: JOHAN PLOEGAERT
VEEHOUDERIJ BV, ZODIAC
GEBOUW 122, DE ELST 1
6708 WD WAGENINGEN
NEDERLAND

CERTIFIED CONCENTRATION

Component Name	Concentration (Moles)	Accuracy (+/-%)
AMMONIA	101, PPM	1
NITROGEN	BALANCE	

TRACEABILITY

Description	Traceability Type	Traceable To
ANALYTICAL TRACEABILITY	GAS STANDARDS	

APPROVED BY: H.F.A. AKKERMANS DATE: 01-05-2015

SUPERVISOR: R.J. Hofmeester

Page 1 of 2

An Air Liquide Group Company

SPECIFICATIONS

Component Name	Requested Concentration (Moles)		Gravimetric Concentration (Moles)	Analyzed Concentration (Moles)		Blend Tolerance Result (+/- %)	Blend Process Accuracy Result (+/- %)	Analytical Accuracy Result (+/- %)	Interlocking Result (+/- %)
AMMONIA	100,	PPM	N/A	101,	PPM	1,0	N/A	1,00	N/A
NITROGEN									

TRACEABILITY

Analytical Traceability

Type/SRM No.	Expiration Date	Cylinder Number	Concentration	Component
SRS TSTD	30Apr2015		0,000	

PHYSICAL PROPERTIES

Cylinder Size: 50L

Pressure: 150 BAR
Expiration Date: 29Apr2018

Valve Connection: DIN 5

Min. Cyl. Pressure: 5 BAR

SPECIAL HANDLING INSTRUCTIONS

If a dew point is listed, do not use or store cylinder at or below the stated dew point temperature. Possible condensation of heavier components could result. In the event the cylinder has been exposed to temperatures at or below the dew point, place the cylinder in heated area for 24 hours and then roll cylinder for 15 minutes to remix.

Use of calibration standards at or below the stated dew point temperature may result in calibration error.

Page 2 of 2

Appendix C - Certificates of Signal 821 diluter

2015:

SIGNAL GROUP

SIGNAL GROUP LIMITED
12 DOMAN ROAD, CAMBERLEY
SURREY, ENGLAND GU15 3DF
TELEPHONE: +44 (0)1276 682841 / 4 (4 LINES)
FAX: +44 (0)1276 691302
E-MAIL: INSTRUMENTS@SIGNAL-GROUP.COM
SITE: WWW.SIGNAL-GROUP.COM

CERTIFICATE OF CALIBRATION

CONSIGNEE Wageningen

ORDER NUMBER

W/O NUMBER

DATE OF CALIBRATION 01/12/2015

DESCRIPTION OF GOODS	SERIAL NO.	REMARKS
821S gas divider	4789	

CALIBRATION RESULTS

DIVIDER SETTING	ACTUAL RESULT
100.00%	100.00
90.00%	90.00
80.00%	79.92
70.00%	69.93
60.00%	60.04
50.00%	50.06
40.00%	39.90
30.00%	30.02
20.00%	20.08
10.00%	10.12
0.00%	0.00

CALIBRATION GAS DETAILS

CALIBRATION GAS TYPE	SUPPLIER	CERTIFICATE No.	TRACEABLITY
1000ppm C3H8/air	BOC	1433495	NPL

SIGNAL AUDIT PROCEDURE NUMBER TPB00101

UNCERTAINTY OF MEASUREMENT +/- 0.2% F.S.D.

Signal Group certifies that the materials and processes used in the calibration of the unit detailed above hereon unless otherwise stated conform in all respects to Signal Group Ltd procedures and specifications

SIGNED Roy Kinslow

2005:

CERTIFICATE OF CALIBRATION

SIGNAL INSTRUMENTS

STANDARDS HOUSE,
12 DOMAN ROAD, CAMBERLEY
SURREY, GU15 3DF, ENGLAND.
TELEPHONE: +44(0)1276 682841/6
FACSIMILE: +44(0)1276 691302
E-MAIL: instruments@signal-group.com
Web: www.signal-group.com

Everything for the Emissions Engineer

CONSIGNEE Agrotechnology & Food Innovations

ORDER NUMBER

W/O NUMBER

DATE OF CALIBRATION 14/10/05

DESCRIPTION OF GOODS	SERIAL No	REMARKS
821S GAS Divider	4789	

CALIBRATION RESULTS

DIVIDER SETTING	EXPECTED RESULT	ACTUAL RESULT
100.00%		100.00
90.00%		89.92
80.00%		79.82
70.00%		69.81
60.00%		59.85
50.00%		50.03
40.00%		39.85
30.00%		30.01
20.00%		20.00
10.00%		10.07
0.00%		0.0

CALIBRATION GAS DETAILS

CALIBRATION GAS TYPE	SUPPLIER	CERTIFICATE No.	TRACEABILITY
1000ppm C3H8 [illegible]	Messer	H114073	NPL

SIGNAL AUDIT PROCEDURE NUMBER TPBOC101

UNCERTAINTY OF MEASUREMENT +/- 0.2% f.s.d.

Signal Group certifies that the materials and processes used in the calibration of the unit detailed above hereon unless otherwise stated conform in all respects to Signal Group Ltd procedures and specifications.

SIGNED

Certificate Number FM 33093 BS EN ISO 9001:2000

A DIVISION OF SIGNAL GROUP LTD. REGISTERED IN ENGLAND NO. 1369865

1996 (used for dataprocessing):

Certificaat

Nederlands Meetinstituut

Nummer 316430-01
blad 1 van 2

Aanvrager:	IMAG - DLO Mansholtlaan 10 - 12 Wageningen
Aangeboden:	Een gas divider fabrikaat : Signal type : 821 serie nummer : 4789 bereik : 0 - 100%, in stappen van 10%
Wijze van onderzoek:	De door de gas divider gemaakte verdunningen zijn onderzocht met een referentiemonitor welke gekalibreerd is met primaire standaard gasmengsels. Als nulgas is hoogzuivere stikstof aangeboden aan de gas divider. Als spangas is een mengsel gebruikt van (1001 ± 10) · 10^{-6} mol/mol stikstofmonoxide in stikstof. Bepaald is het werkelijke verdunningspercentage. De metingen werden uitgevoerd bij een omgevingstemperatuur van 21 ± 0,5 ° C.
Datum van onderzoek:	6 tot en met 20 september 1996
Resultaat:	De resultaten staan vermeld op blad 2 van 2. De totale meetonzekerheid is gebaseerd op twee maal de standaardafwijking (2s).
Herleidbaarheid:	De bij de kalibratie gebruikte gasmengsels zijn herleidbaar naar de primaire standaard gasmengsels stikstofmonoxide in stikstof.
Datum en ondertekening:	24 september 1996 NMi Van Swinden Laboratorium B.V. R. M. Wessel Afdeling Chemie

Nederlands Meetinstituut
Postbus 654
2600 AR Delft (NL)
Schoemakerstraat 97
2628 VK Delft
Telefoon (015) 269 15 00
Telefax (015) 261 29 71

Nederlands Meetinstituut N.V. (Ingeschreven bij de Kamer van Koophandel Delft onder nummer 28701)

Werkmaatschappijen:
NMi Certin B.V. (KvK nr. 33418)
NMi Van Swinden Laboratorium B.V. (KvK nr. 28703)
NMi IJkwezen B.V. (KvK nr. 28700)
NMi International B.V. (KvK nr. 39176)

Dit certificaat wordt verstrekt onder het voorbehoud dat generlei aansprakelijkheid wordt aanvaard en dat aanvrager vrijwaring geeft voor elke aansprakelijkheid jegens derden.

Reproduktie van het volledige certificaat is toegestaan. Gedeelten van dit certificaat mogen slechts worden gereproduceerd na verkregen schriftelijke toestemming.

Form. 962

Nederlands Meetinstituut

Nummer 316430-01
blad 2 van 2

Resultaat: Hieronder wordt het resultaat van de kalibratie weergegeven.

Meetbereik 0-100% in stappen van 10%.

Werkelijk verdunnings-percentage (%)	ingesteld verdunnings-percentage (%)
10,05 ± 0,10	10
19,91 ± 0,12	20
29,86 ± 0,12	30
39,73 ± 0,18	40
49,93 ± 0,20	50
60,04 ± 0,25	60
69,88 ± 0,25	70
79,95 ± 0,25	80
89,99 ± 0,25	90

NMi Van Swinden Laboratorium B.V.

Form. 962

Appendix D - Certificate of EPM 797-430 diluter

WAGENINGEN UR
For quality of life

KALIBRATIE
RvA K 029

Kalibratie-certificaat

C02009-3

Aanvrager: ASG Veehouderij B.V.
Postbus 65
8200 AB Lelystad

Aangeboden: Laboratoriumverdunner
Fabrikaat: EPM
Type: 797-430
Serienummer: E 77

Wijze van onderzoek: Het onderzoek werd uitgevoerd met CO als tracergas. De CO-concentraties werden gemeten met een gekalibreerde GFC CO-analyser. De verdunningen werden berekend uit de in en uitgaande CO-gasconcentraties van het verdunningstoestel.

Omgevingscondities Het onderzoek is uitgevoerd bij een ruimtetemperatuur van (21 ± 2) °C.

Datum van onderzoek: 17 maart 2009.

Resultaat: De resultaten en meetonzekerheden staan vermeld op blad 2.

Onzekerheid: De gerapporteerde onzekerheid is gebaseerd op de standaardonzekerheid vermenigvuldigd met een dekkingsfactor $k = 2$, welke overeenkomt met een betrouwbaarheids-interval van ongeveer 95%. De standaardonzekerheid is bepaald volgens EA-4/02.

Herleidbaarheid: De metingen zijn uitgevoerd met meetmiddelen waarvan de herleidbaarheid naar (inter)-nationale standaarden is aangetoond ten overstaan van de Raad voor Accreditatie.

Datum en ondertekening: Wageningen, 26 maart 2009

Ing. J.P.M. Ploegaert
Coördinator Geurlaboratorium

WAGENINGEN UR
Certificate : C02009-3
Date of issue : 26-03-2009
Date of last calibration : 17-03-2009
Recalibration before :

Versie 01-05-2007

Wageningen UR
ASG Veehouderij B.V.
Cluster MHE Wageningen
Bornsesteeg 59
6708 PD Wageningen
Telefoon +31 (0) 317 487276
Telefax +31 (0) 317 475347

1/2

WAGENINGEN UR
For quality of life

KALIBRATIE
RvA K 029

Kalibratie-certificaat

C02009-3

Instelling	Verdunning		Onzekerheid (k=2)	Nauwkeurig-heid	Stabiliteit %
	[-]	[-]	[-]	[-]	[-]
2.6	10.0	+/-	0.03	0.008	0.24
3.0	11.0	+/-	0.01	0.008	0.28
4.0	13.5	+/-	0.06	0.005	0.22
5.0	16.0	+/-	0.08	0.004	0.19
6.0	18.7	+/-	0.15	0.003	0.23

Opmerkingen: Metingen conform NEN-EN 13725:2003. Uitgevoerd door JP.
De laboratoriumverdunner werd met de hand bediend.

Versie 01-05-2007

Wageningen UR
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2/2

To explore the potential of nature to improve the quality of life

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E info.livestockresearch@wur.nl
www.wageningenUR.nl/livestockresearch

Together with our clients, we integrate scientific know-how and practical experience to develop livestock concepts for the 21st century. With our expertise on innovative livestock systems, nutrition, welfare, genetics and environmental impact of livestock farming and our state-of-the art research facilities, such as Dairy Campus and Swine Innovation Centre Sterksel, we support our customers to find solutions for current and future challenges.

The mission of Wageningen UR (University & Research centre) is 'To explore the potential of nature to improve the quality of life'. Within Wageningen UR, nine specialised research institutes of the DLO Foundation have joined forces with Wageningen University to help answer the most important questions in the domain of healthy food and living environment. With approximately 30 locations, 6,000 members of staff and 10,000 students, Wageningen UR is one of the leading organisations in its domain worldwide. The integral approach to problems and the cooperation between the various disciplines are at the heart of the unique Wageningen Approach.