

PERFORMANCE ANALYSIS OF LOW-COST GAS SENSORS FOR AIR QUALITY CONTROL



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Fixed/indicative measurements: definition

'fixed measurements' measurements taken at fixed sites, to determine the levels in accordance with the relevant *Data Quality Objectives (DQO)*;

'indicative measurements' measurements which meet *DQOs* that are less strict than those required for *fixed measurements*;

They are use to check at *Limit Values* for an *Averaging Time* (for ozone 60 ppb – mean of 8 *hourly values*)

European DIRECTIVE 2008/50/EC on ambient air quality and cleaner air for Europe, art. 2

Barcelona, 20 June 2013

AQD: Data Quality Objectives (DQO)

	SO ₂ , NO ₂ /NO/ NO _x , CO	Benzene	O ₃
Uncertainty for fixed measurements	15 %	25 %	15 %
Uncertainty for indicative measurements	25 %	30 %	30 %

Indicative methods: what for ?

Fixed measurements are mandatory in zones and agglomerations where the upper assessment thresholds are exceeded, otherwise indicative methods can be used.

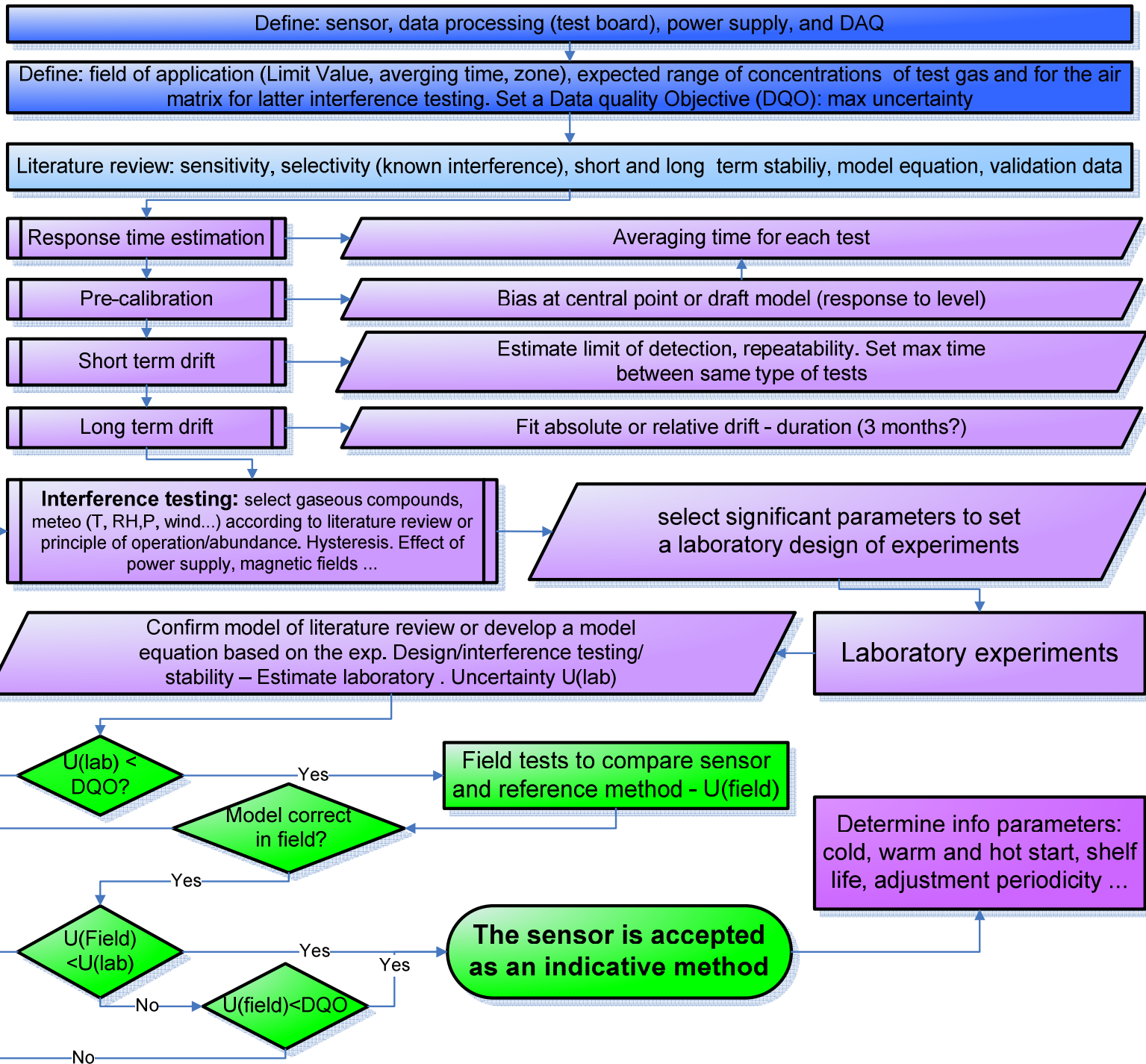
The use of indicative measurements allows for reduction of 50 % of the required minimum number of fixed sampling points.

European DIRECTIVE 2008/50/EC on ambient air quality and cleaner air for Europe, art. 6

An indicative method:

Only parameter needed to demonstrate that a method is suitable as an indicative method is its measurement **uncertainty** ...

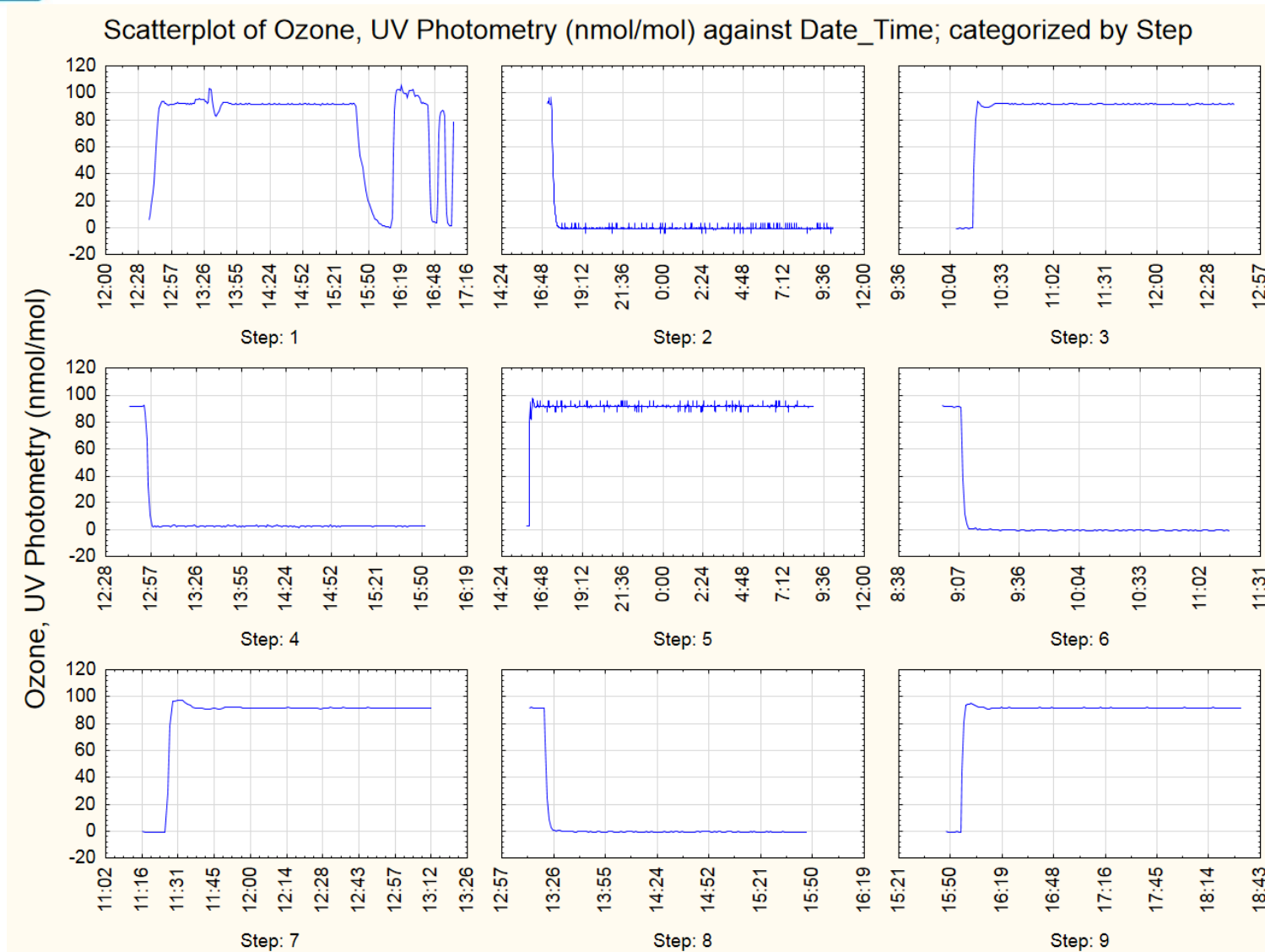
However, to estimate the measurement uncertainty of micro-sensors, a huge quantity of metrological parameters has to be determined ...



	Tests	Temperature, °C	Relative humidity, %	Comment
1	Response Time	Mean	Mean	Three times: 0 to 80 % of Full Scale and 80% of FS to 0
2	Pre-calibration	Mean	Mean	At least 3 levels including 0, LV, IT, AT, CL, LAT and UAT
3-1	Repeatability	Mean	Mean	0 and 90 % of LV, 3 repetitions every averaging time
3-2	Short term drift	Mean	Mean	0, 50 % and 80 % of LV, 3 repetitions every 24 hours
3-3	Long term drift	Mean	Mean	0, 50 % and 80 % of LV, repeated every 2 weeks during 3 months
4-1	Air matrix	Mean	Mean	Filtered air, laboratory air and ambient air at 0 and LV
4-2	Gaseous interference	Mean	Mean	Test selected interferences at zero and average level in ambient air
4-3	Temperature	From mean-10 °C to mean+10 °C by step of 5 °C	Mean	At LV
4-4	Humidity	Mean	From mean-20% to mean+20% by step of 10%	At LV
4-5	Hysteresis	Mean	Mean	Increasing-decreasing-increasing concentration of the pre-calibration levels
4-6	Pressure	Mean	Mean	overpressure 10 mbar and under pressure 5 mbar
4-7	Power supply effect	Mean	Mean	At LV test under 210, 220 and 230 V
4-8	Wind velocity	Mean	Mean	from 1 to 5 m/s (needed?)
4-1	Lab experiments (model)	Mean-10°C, mean, mean -10°C	Mean-20%, mean, mean + 20 %	0, LV, AT for each significant parameters: temperature and humidity (levels) and wind, pressure and interference (2 levels)
4-2	Field experiments			At an automatic station equipped with reference method of measurements
5-1	Cold start, warm start, hot start	Mean	Mean	At LV

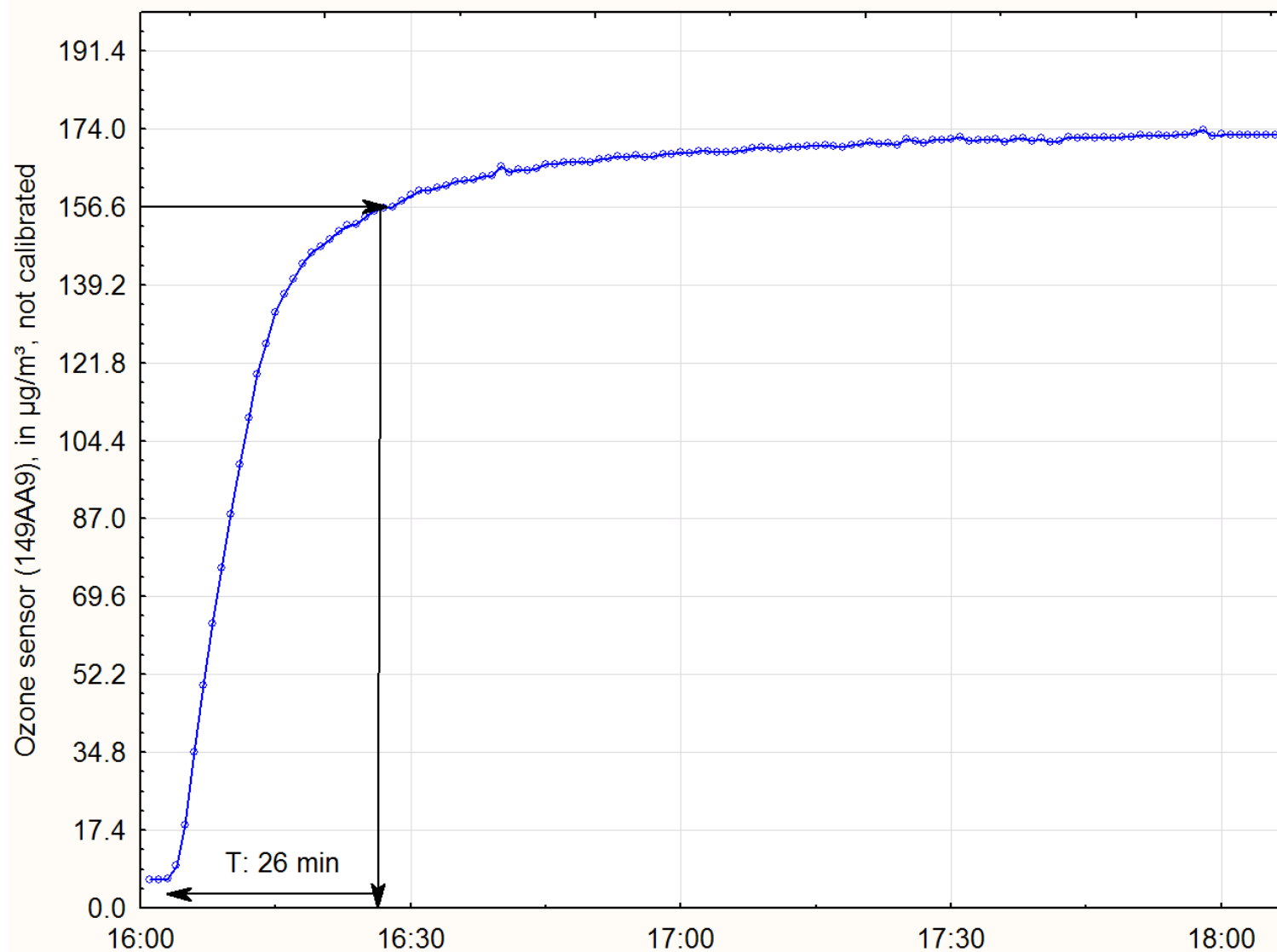


1 - Response time





1 - Response time





1 - Response time



	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8	Step 9
O ₃ , nmol/mol	-0.6±0.2	91.7±0.2	2.6±0.3	91.7±0.2	-0.5±0.2	91.7±0.3	-0.5±0.2	91.±0.2
NO ₂ , nmol/mol	0.7±0.1	0.7±0.2	1.0±0.1	0.7±0.1	0.7±0.1	0.7±0.2	0.7±0.1	0.7±0.2
NO, nmol/mol	1.7±0.1	1.8±0.1	1.8±0.1	1.7±0.1	1.8±0.1	1.8±0.1	1.8±0.1	1.8±0.1
Temperature, °C	22.0±0.1	22.1±0.0	22.1±0.0	22.0±0.0	22.0±0.0	22.0	22.0	22.0
Humidity, %	57.7±0.7	60.0±0.0	59.9±0.1	60.0±0.0	60.0±0.0	60.0±0.1	60.0±0.2	60.0±0.0
Pressure, kPa	996±2	999±0.1	998±0.1	1000±0.3	1001±0.1	1000±0.2	999±0.4	998±0.1
Time length	900'	210'	210'	1050'	195'	180'	150'	165'

	Step 2	Step 3	Step 4 long 90	Step 5	Step 6	Step 7	Step 8	Step 9
	Fall	Rise	Fall	Rise	Fall	Rise	Fall	Rise
t ₉₀ , O ₃ UV Photometry	14 min	4 min	4 min	5 min	4 min	4 min	3 min	3 min
t ₉₀ , CairClipO ₃	5 min	3 min	4 min	4 min	4min	6 min	4 min	4 min
Response time, 4 x t ₉₀	20 min	12min	16 min	16 min	16 min	24 min	16 min	16 min



1 Response time



	Rise Time (n=3)	Fall Time (n=3)	Average Time	Response time per type
Chamber – UV analyser (subtracted)	3'	5'	4'	
Unitec, Sens3000 (n =2)	47'	75'	61'	31'
e2v, MICS-2610 (n =2)	1'	12'	6'	
FIS, SP-61(n =2)	57'	54'	56'	
Ingenieros Assessores, NanoEnvi (n =1)	8'	14'	11'	
e2v, OMC3 (n=2)	-	-	-	
e2v, OMC2 (n=2)	-	-	-	
e2v, MICS_Oz47 (n =7)	19'	27'	23'	
IMN2P, WO3 (n =2)	8'	16'	12'	12'
Sensoric, O3E1F + Test Panel (n =1)	1'	2'	2'	3'
Sensoric, O3E1 + 4-20mA Board (n =2)	4'	5'	4'	
αSense B4 (n =2)	1'	2'	2'	
CairclipO3NO2 (n=1)	3'	3'	3'	
Sensors' Average response Time	15'	21'	18'	



1 - Response time

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- Response time $<$ the averaging time of the Limit Value of the Directive (mean of hourly values over 8 hours). Duration of tests.
- Similar response time for rise and fall cycles?
- The sensor is fast enough for areas where air pollutants change fast (a few minutes) or for mobile measurements?

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2 Pre-calibration

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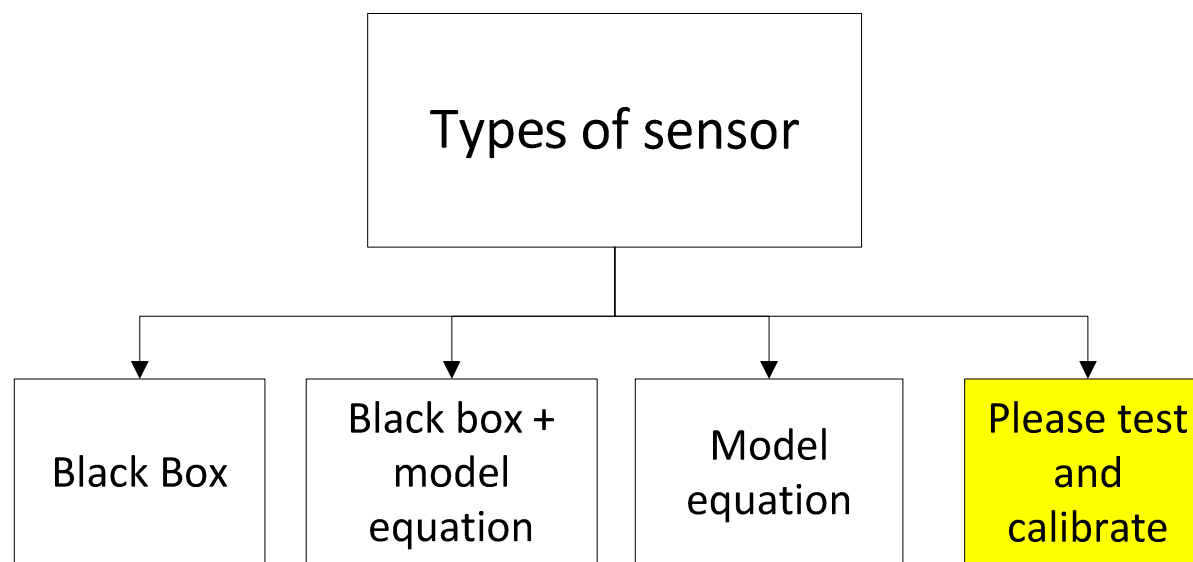


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- Make sure that the transformation of sensor responses into gaseous concentrations does not include any bias at the mean temperature and relative humidity.

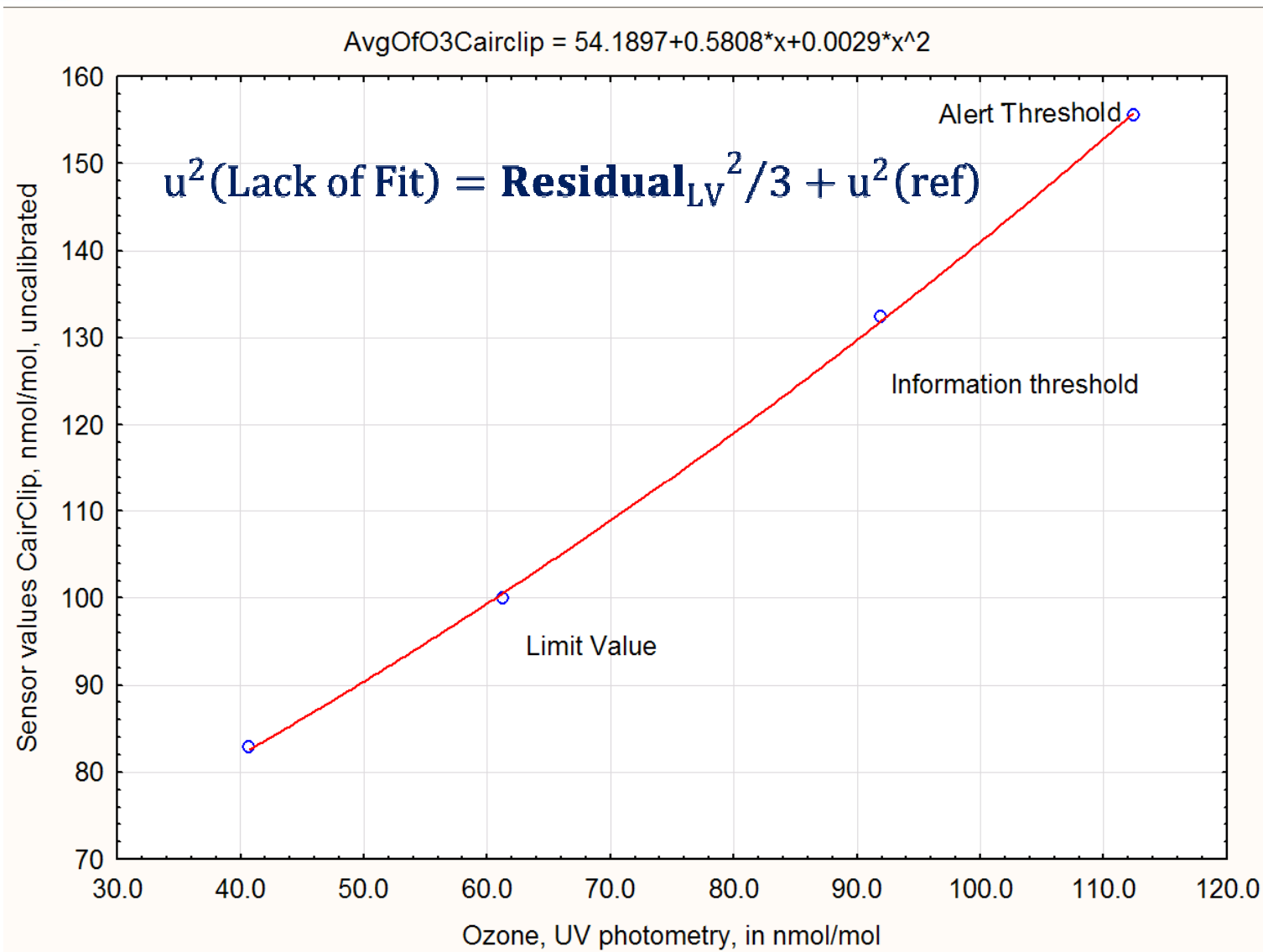


2 Pre-calibration



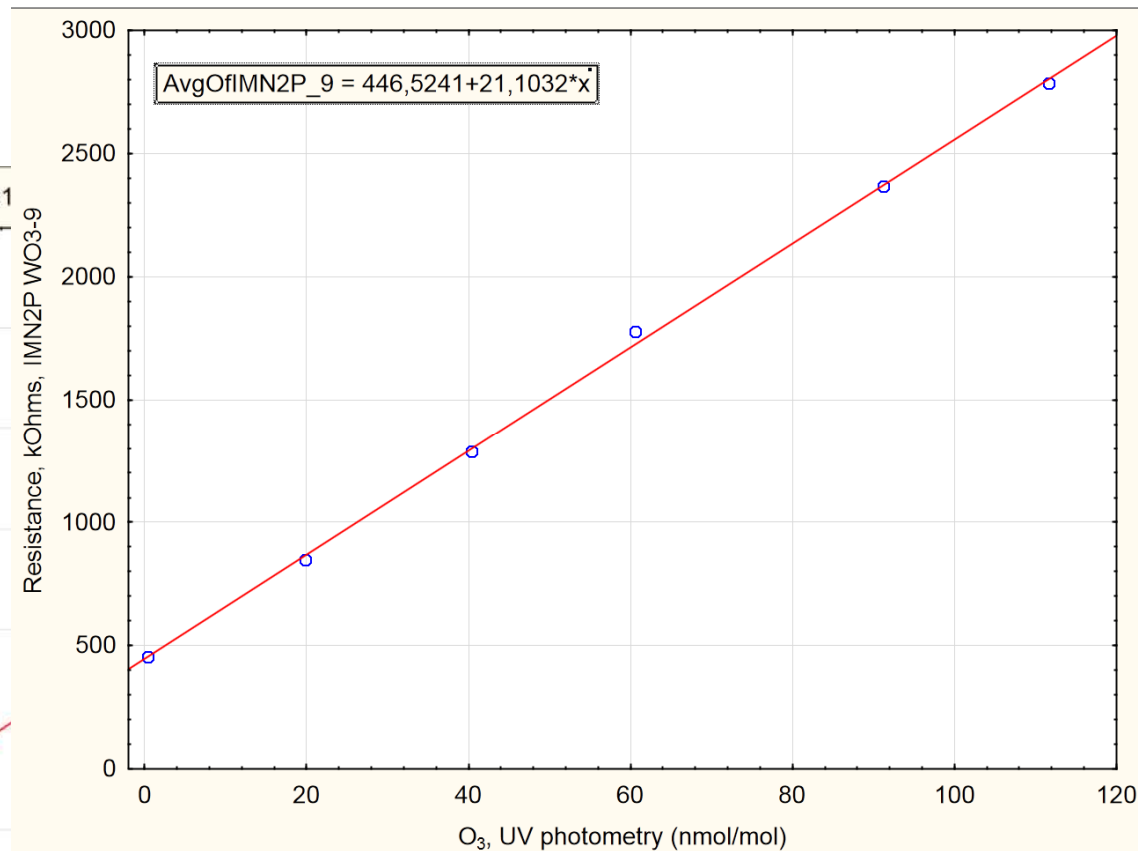
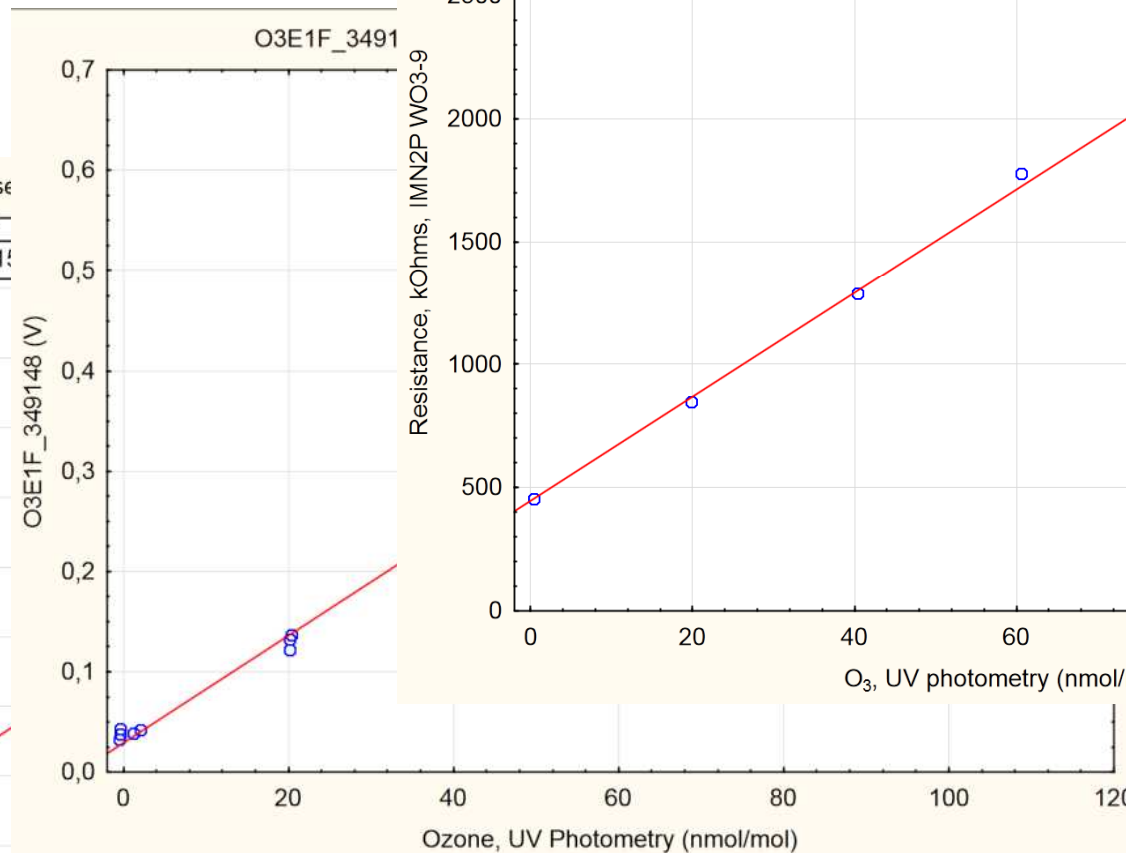
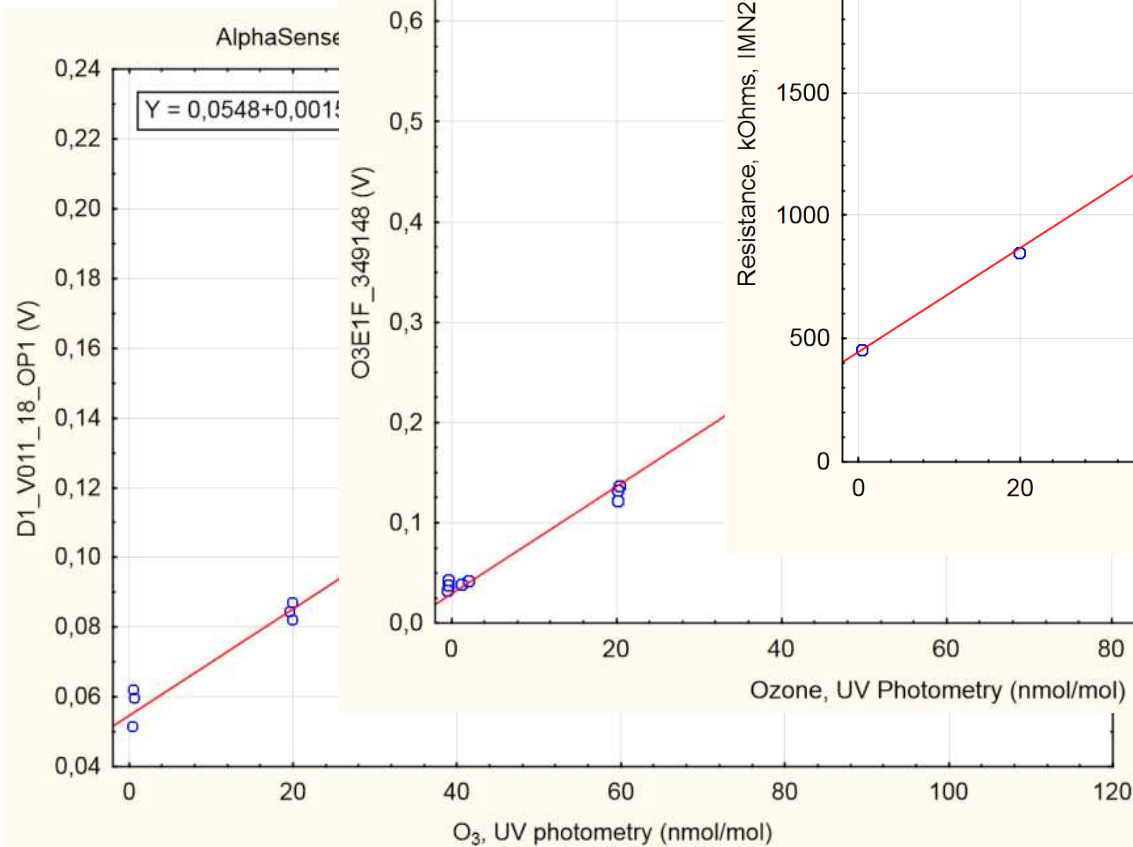


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4-8	Wind velocity	Mean	Mean	from 1 to 5 m/s (needed?)
4-1	Lab experiments (model)	Mean-10°C, mean, mean -10°C	Mean-20%, mean, mean + 20 %	0, LV, AT for each significant parameters: temperature and humidity (levels) and wind, pressure and interference (2 levels)
4-2	Field experiments			At an automatic station equipped with reference method of measurements
	Cold start warm start hot			



3.1 Repeatability (hourly values)



	O3	NO2	NO	CO	Temp	Humidity	Pressure	CairClipO3
	ppb	ppb	ppb	ppb	°C	%	hPa	ppb
Mean ± s (n=9)	0,6 ± 0.1	0,7 ± 0.0	1,7 ± 0.1	250,2 ± 13	22,0 ± 0,0	59,0 ± 0,9	994,3 ± 3,9	38.2 ± 2.9
Mean ± s (n=13)	91,7 ± 0.0	0,7 ± 0.0	1,7 ± 0.0	268,2 ± 13	22,0 ± 0.0	60,0 ± 0.0	1000,3 ± 0,5	90.0 ± 0.6

- **Repeatability: 1.6 ppb ($2\sqrt{2}s$ at 90 ppb of O3) possible difference between two hourly values :.**
- **Limit of detection: 5.8 ppb (3s of O3 at 0 ppb)**
- **Limit of quantification: 29 ppb (3s of O3 at 0 ppb).**
- **Sensor artificially blocked at minimum at about 40 ppb. Limits of detection and limit of quantification overestimated**

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5-1	Cold start, warm start, hot start	Mean	Mean	At LV



3.2 Short term stability

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hourly averages after 1 day drift

$$D_s = \frac{\sum_{After} R_s}{n} - \frac{\sum_{Before} R_s}{n}$$

$$D_s (\%) = \left| 1 - \frac{\sum_{After} R_s}{\sum_{Before} R_s} \right|$$



1 day drift for hourly averages at 0,60 and 90 nmol/mol

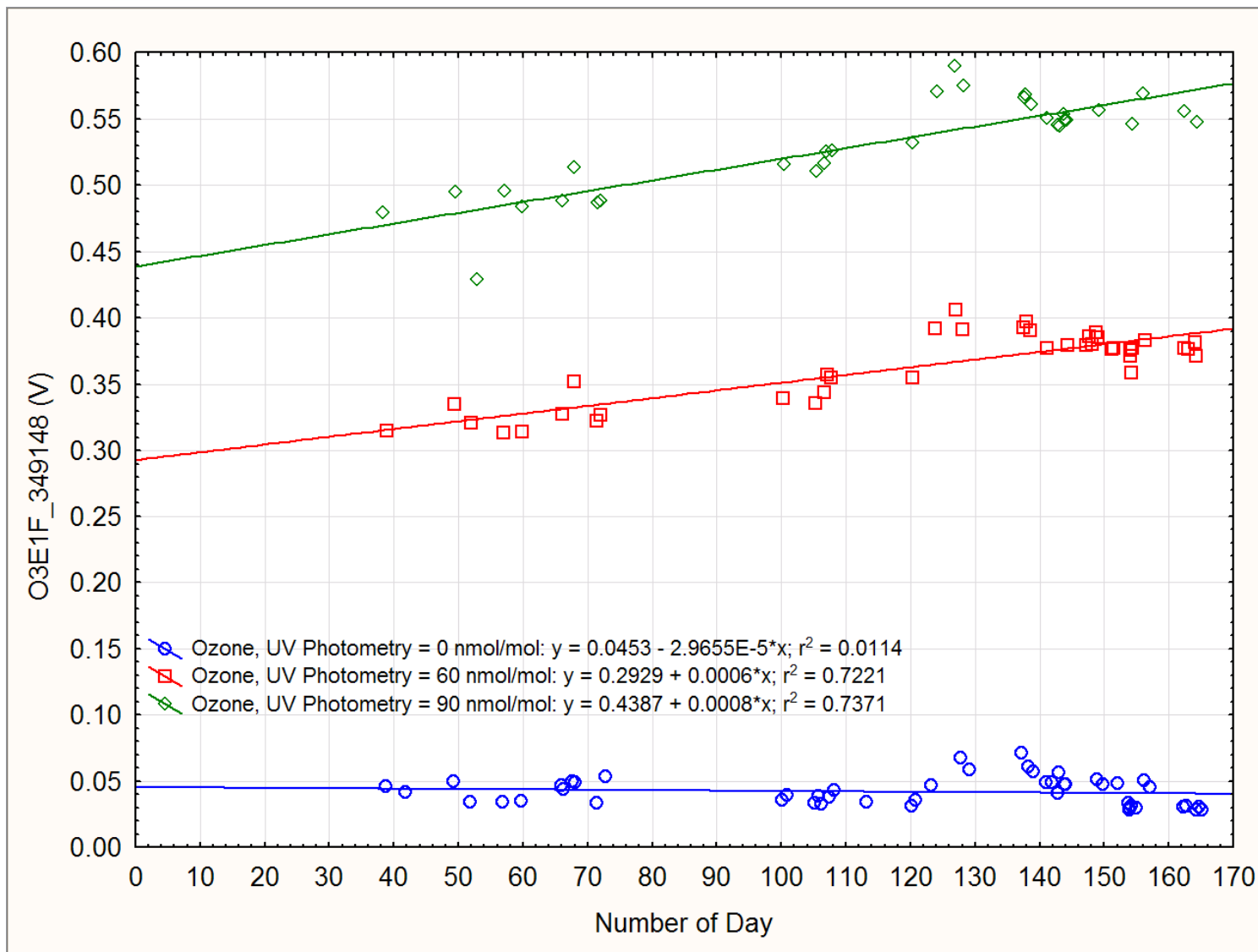


	Ds0, nmol/mol (n~19)	Ds60, nmol/mol- % (n~8)	Ds90, nmol/mol- % (n~6)
Res 1 (n =2)	5.4	3.1 – 1.9 %	2.7 – 2 %
Res 2 (n =2)	On going data treatment		
Res 3 (n =2)	13	15 – 9.5 %	15 – 8.9 %
Res 4 (n =2)	1.8	2.3 – 3.6 %	2.1 – 2.6 %
Res 5 (n=2)	On going data treatment		
Res 6 (n=2)	On going data treatment		
Res 7 (n =1)	1.9	2.9 – 2.5 %	2.8 – 2.0 %
Res 8 (n =2)	1.0	2.0 – 2.4 %	4.6 – 4.4 %
Chem 1 (n =1)	1.3	1.2 – 1.8 %	1.2 – 1.1 %
Chem 2 (n =2)	Malfunction	Malfunction	Malfunction
Chem 3 (n = 2)	4.4	1.4 – 0.9 %	0.9 – 0.5 %
Chem 4 (n=1)	4.7	1.0 – 2.5 %	1.5 – 1.6 %

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4-7	Power supply effect	Mean	Mean	At LV test under 210, 220 and 230 V
4-8	Wind velocity	Mean	Mean	from 1 to 5 m/s (needed?)
4-1	Lab experiments (model)	Mean-10°C, mean, mean -10°C	Mean-20%, mean, mean + 20 %	0, LV, AT for each significant parameters: temperature and humidity (levels) and wind, pressure and interference (2 levels)
4-2	Field experiments			At an automatic station equipped with reference method of measurements
5-1	Cold start, warm start, hot start	Mean	Mean	At LV



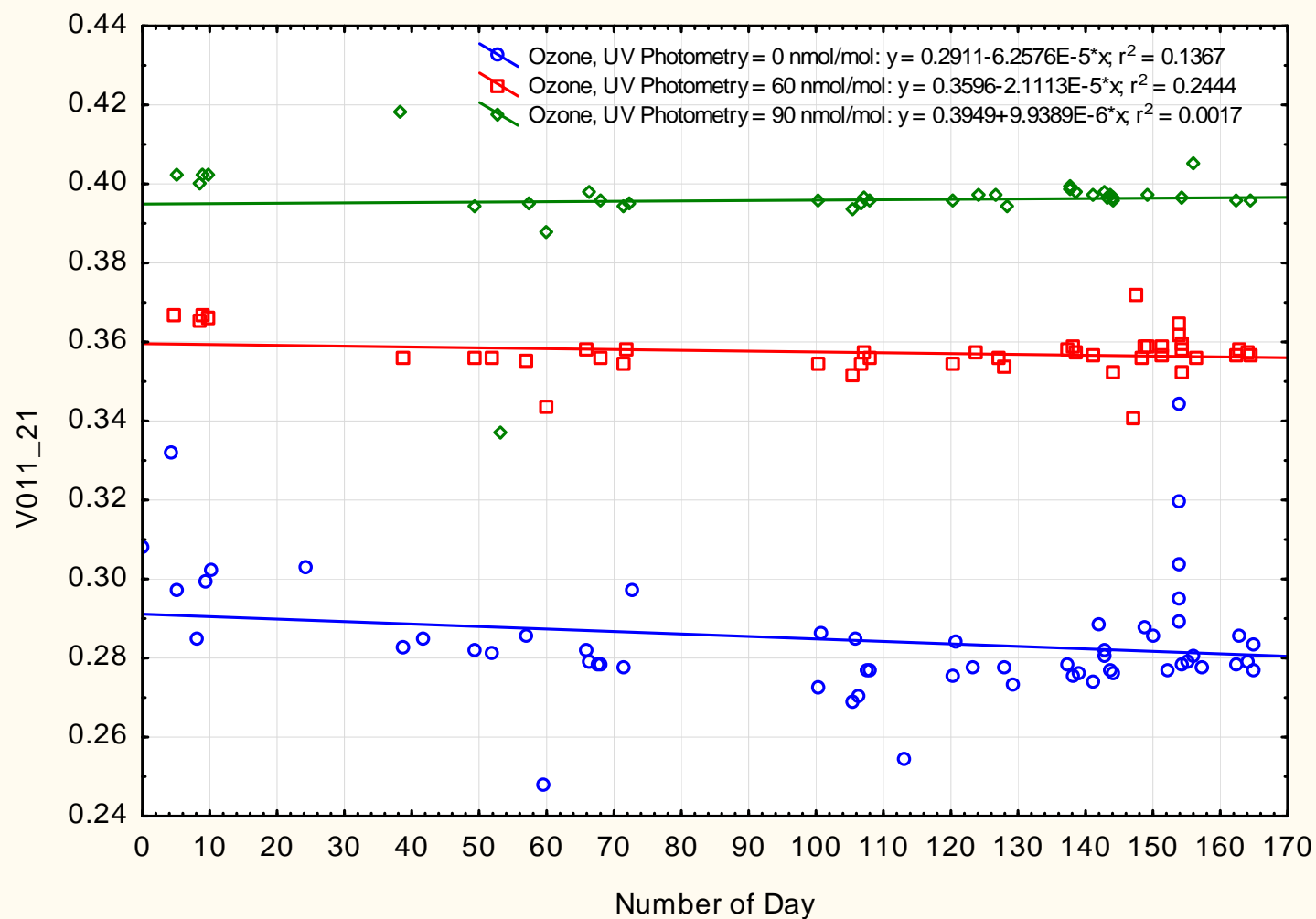
3.3 Long term stability! 150 days



Long term stability! 150 days

a)

Scatterplot of V011_21 (V)





3.3 Long term stability



- Identify trends in the long-term stability plots
- Decide if a correction can be added in the sensor model equation
- Estimate $u(D_{ls})$, the contribution to the lab uncertainty: drift + noise (lack of fit)
- Set a periodicity of re-calibration of the sensor (a way to decrease the drift over time)
- Shall be used to correct the drift of sensors in the test results of this protocol





3.2 Stability 150 days, hourly averages at 0, 60 and 90 ppb



	Regression line		
	Ds0, nmol/mol/100day	Ds60, nmol/mol/100day	Ds90, nmol/mol/100day
Res 1 (n =2)	3	9 – 15 %	13 – 15 %
Res 2 (n =2)	On going data treatment		
Res 3 (n =2)	3	29 – 50 %	34 – 35 %
Res 4 (n =2)	3	4 - 7 %	5 -
Res 5 (n=2)	On going data treatment		
Res 6 (n=2)	On going data treatment		
Res 7 (n =1)	On going data treatment		
Res 8 (n =2)	On going data treatment		
Chem 1 (n =1)	-1	11 – 20 %	15 – 17 %
Chem 2 (n =2)	Malfunction	Malfunction	Malfunction
Chem 3 (n = 2)	-6	-3 – 5 %	1 – 1 %
Chem 4 (n=1)	On going data treatment		

+ Noise

	Tests	Temperature, °C	Relative humidity, %	Comment
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4.2 Gaseous interference

O₃ sensors

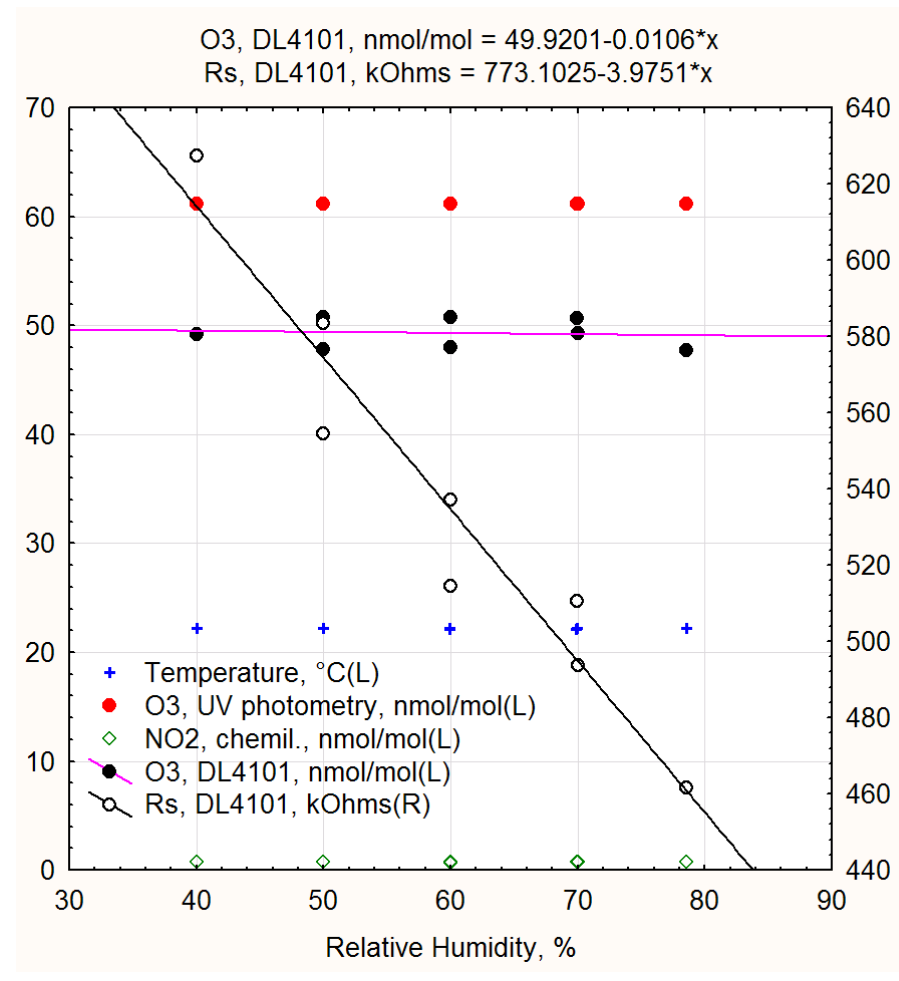
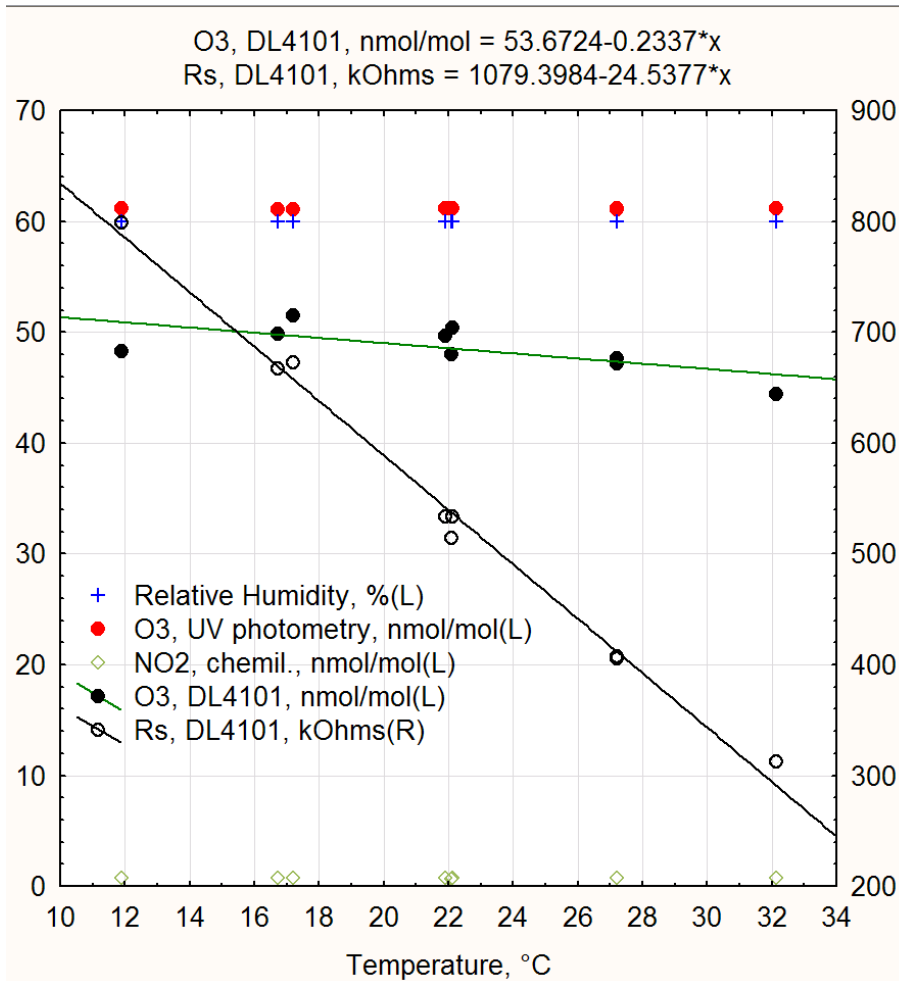


Sensors/	NO ₂	NO	CO	CO ₂	NH ₃
Interference, nmol/mol	100	100	8000	Purified air	± 85
O ₃ , nmol/mol	60	0	60	60	60
RH (%)	60 %	60 %	60 %	60 %	60 %
T (°C)	22 °C	22 °C	22 °C	22 °C	22 °C
Res 1 (n =2)	14.1 %	-5.3 %	9.8 %	-6.2 %	6.0 %
Res 2 (n =2)	-3.3 %	-	3.6 %	-0.1 %	-0.3 %
Res 3 (n =2)	14.5 %	-10 %	13.4 %	5.0 %	18 %
Res 4 (n =2)	12.6 %	-1.6 %	-8.9 %	1.0 %	3.5 %
Res 5 (n=2)	-	-	-	-	-
Res 6 (n=2)	-	-	-	-	-
Res 7 (n =1)	-	-	-	-	-
Res 8 (n =2)	1.2 %	-0.8 %	0.1 %	-0.2 %	-1.9 %
Chem 1 (n =1)	89.3 %	1.3 %	-0.9 %	0.2 %	1.2 %
Chem 2 (n =2)	-	-	-	-	-
Chem 3 (n = 2)	33.7 %	-7.7 %	-1.2 %	-0.4 %	0.1 %
Chem 4 (n=1)	107.7 %	-1.5 %	-2.4 %	0.4 %	2.3 %

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4.3-4.4 Temperature humidity



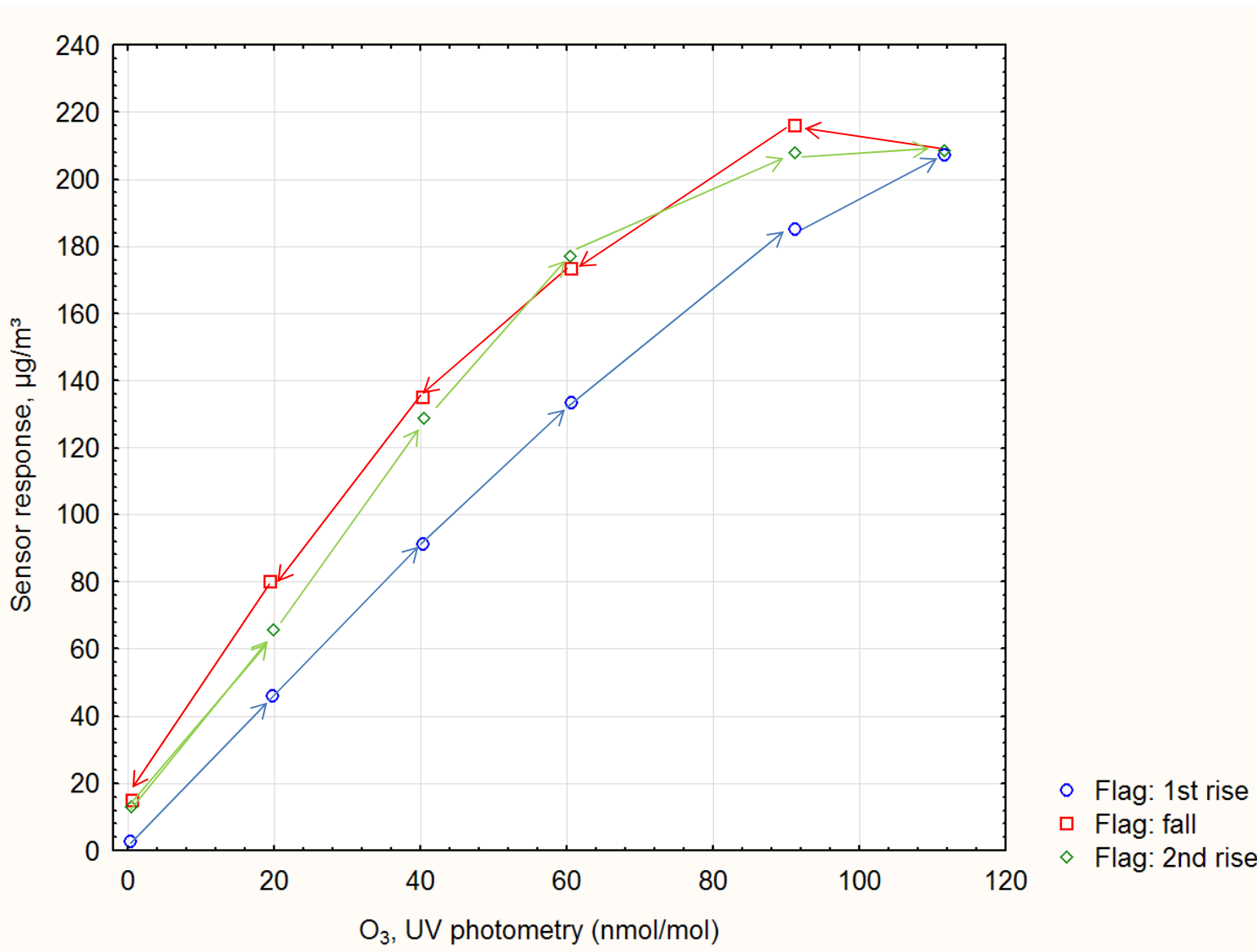
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4-3	Temperature	From mean-10 °C to mean+10 °C by step of 5 °C	Mean	At LV
4-4	Humidity	Mean	From mean-20% to mean+20% by step of 10%	At LV
4-5	Hysteresis	Mean	Mean	Increasing-decreasing-increasing concentration of the pre-calibration levels
4-6	Pressure	Mean	Mean	overpressure 10 mbar and under pressure 5 mbar
4-7	Power supply effect	Mean	Mean	At LV test under 210, 220 and 230 V
4-8	Wind velocity	Mean	Mean	from 1 to 5 m/s (needed?)
4-1	Lab experiments (model)	Mean-10°C, mean, mean -10°C	Mean-20%, mean, mean + 20 %	0, LV, AT for each significant parameters: temperature and humidity (levels) and wind, pressure and interference (2 levels)
4-2	Field experiments			At an automatic station equipped with reference method of measurements
	Cold start warm start hot			



4.5 Hysteresis



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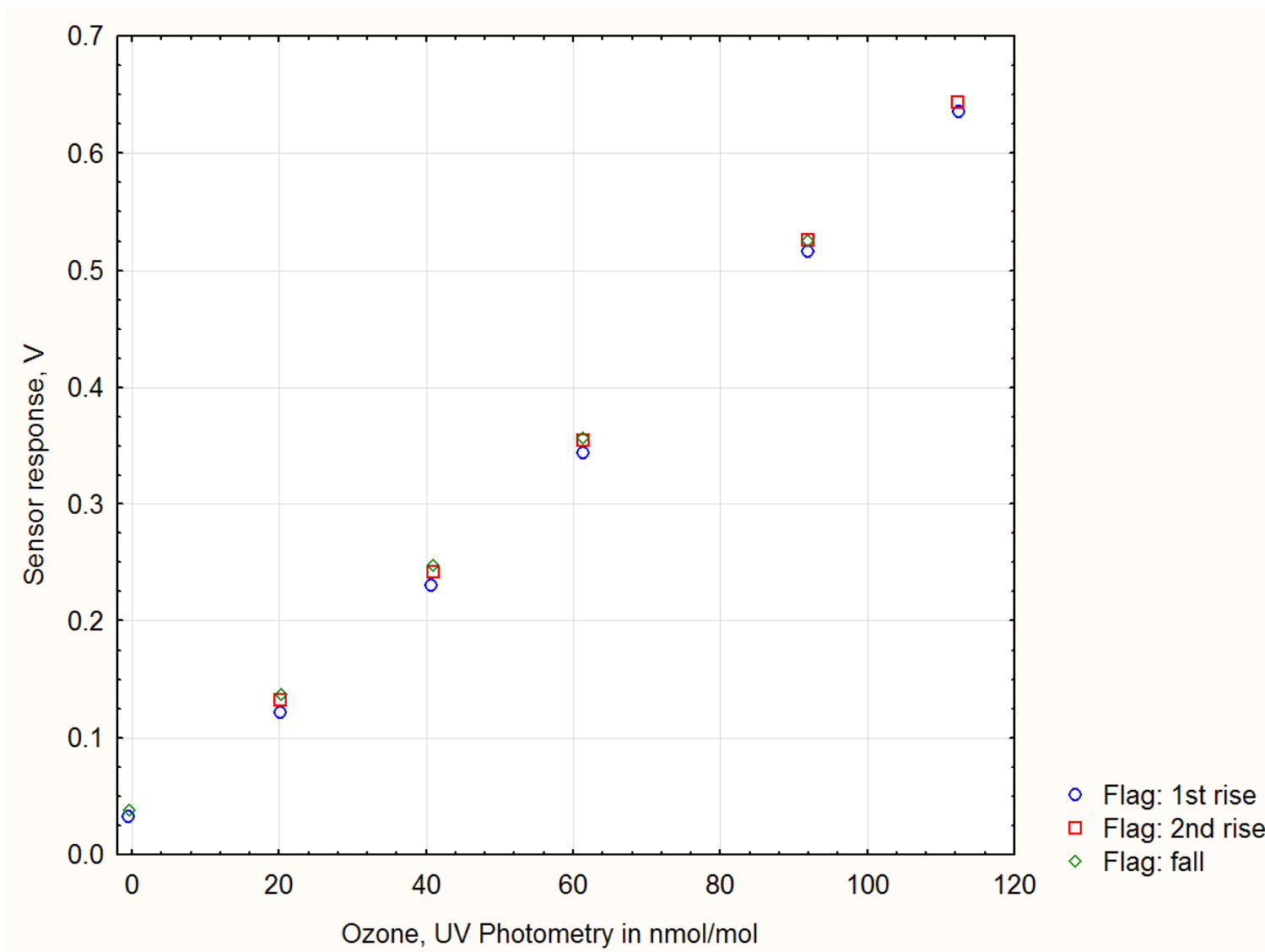
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4.5 Hysteresis



	Tests	Temperature, °C	Relative humidity, %	Comment
1	Response Time	Mean	Mean	Three times: 0 to 80 % of Full Scale and 80% of FS to 0
2	Pre-calibration	Mean	Mean	At least 3 levels including 0, LV, IT, AT, CL, LAT and UAT
3-1	Repeatability	Mean	Mean	0 and 90 % of LV, 3 repetitions every averaging time
3-2	Short term drift	Mean	Mean	0, 50 % and 80 % of LV, 3 repetitions every 24 hours
3-3	Long term drift	Mean	Mean	0, 50 % and 80 % of LV, repeated every 2 weeks during 3 months
4-1	Air matrix	Mean	Mean	Filtered air, laboratory air and ambient air at 0 and LV
4-2	Gaseous interference	Mean	Mean	Test selected interferences at zero and average level in ambient air
4-3	Temperature	From mean-10 °C to mean+10 °C by step of 5 °C	Mean	At LV
4-4	Humidity	Mean	From mean-20% to mean+20% by step of 10%	At LV
4-5	Hysteresis	Mean	Mean	Increasing-decreasing-increasing concentration of the pre-calibration levels
4-6	Pressure	Mean	Mean	overpressure 10 mbar and under pressure 5 mbar
4-7	Power supply effect	Mean	Mean	At LV test under 210, 220 and 230 V
4-8	Wind velocity	Mean	Mean	from 1 to 5 m/s (needed?)
4-1	Lab experiments (model)	Mean-10°C, mean, mean + 10°C	Mean-20%, mean, mean + 20 %	0, LV, AT for each significant parameters: temperature and humidity (levels) and wind, pressure and interference (2 levels)
4-2	Field experiments			At an automatic station equipped with reference



4.1 Experimental design

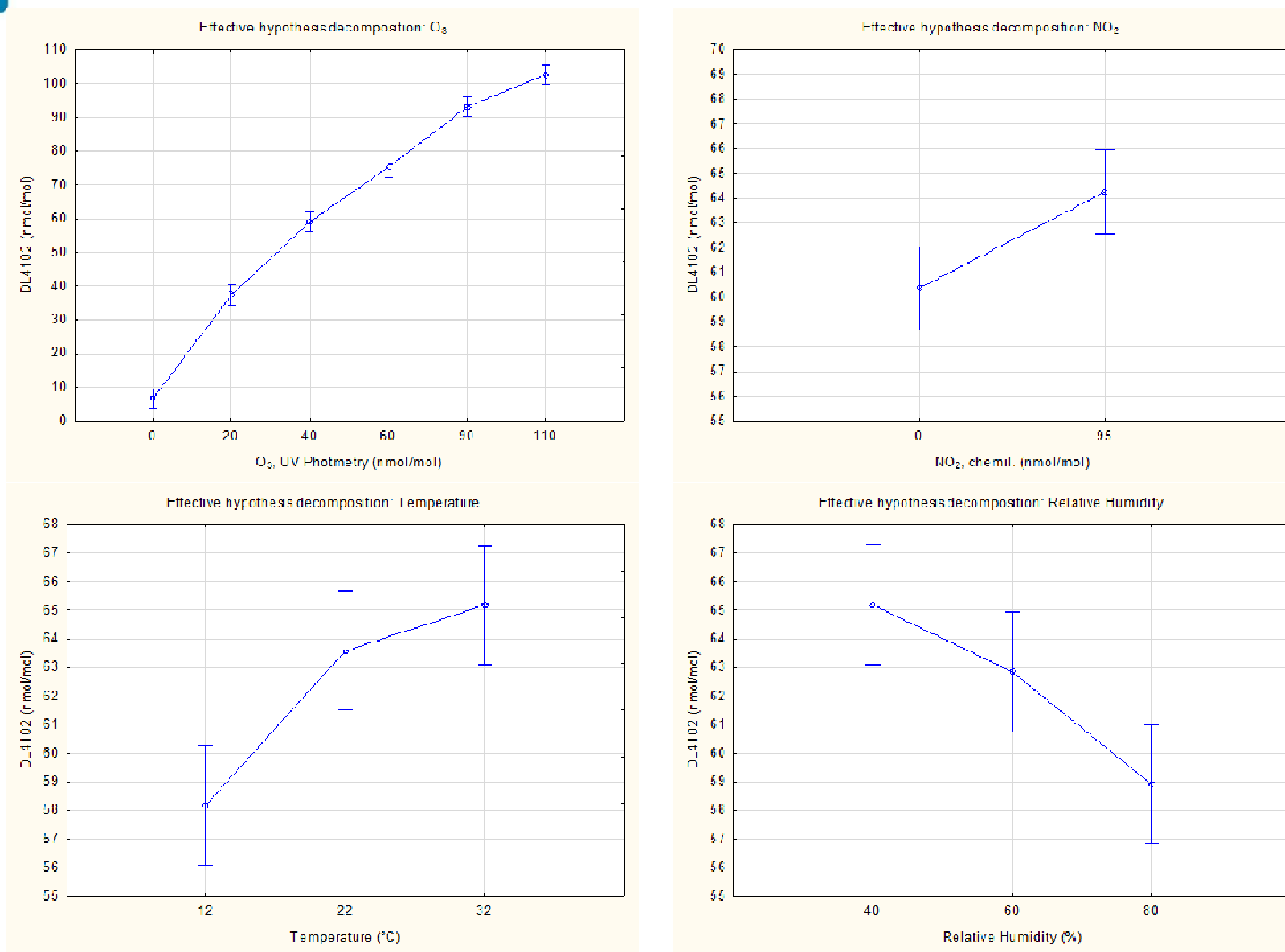
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O_3 nmol/mol	NO_2 nmol/mol	Temp.	Rel. Hum.	Total
0				
20				
40				
60		12	40	
90	0	22	60	
110	100	32	80	
6 levels	2 levels	3 levels	3 levels	108 trials

4.1 Experimental design



$$Sr_{DL4102} = 17.3_{\pm 4.5} + 0.84_{\pm 0.02} * O3 + 0.36_{\pm 0.11} * T - 0.16_{\pm 0.06} * RH + 0.039_{\pm 0.024} * NO2$$



4.1 Lab uncertainty



Using the model equation developed with the desing of experiments design by application of the GUM method.

$$U^2(O3) = 2. \sum \left(\frac{\partial O3}{\partial X_i} \right)^2 u^2(X_i)$$

Add in a quadratic way uncertainty of :

- long-term stability (with re-calibration)
- hysteresis (difficult to include in model equation)
- the interferences that were not included in the model and the ones that cannot be corrected in field

$U < 30 \%$



	Tests	Temperature, °C	Relative humidity, %	Comment
1	Response Time	Mean	Mean	Three times: 0 to 80 % of Full Scale and 80% of FS to 0
2	Pre-calibration	Mean	Mean	At least 3 levels including 0, LV, IT, AT, CL, LAT and UAT
3-1	Repeatability	Mean	Mean	0 and 90 % of LV, 3 repetitions every averaging time
3-2	Short term drift	Mean	Mean	0, 50 % and 80 % of LV, 3 repetitions every 24 hours
3-3	Long term drift	Mean	Mean	0, 50 % and 80 % of LV, repeated every 2 weeks during 3 months
4-1	Air matrix	Mean	Mean	Filtered air, laboratory air and ambient air at 0 and LV
4-2	Gaseous interference	Mean	Mean	Test selected interferences at zero and average level in ambient air
4-3	Temperature	From mean-10 °C to mean+10 °C by step of 5 °C	Mean	At LV
4-4	Humidity	Mean	From mean-20% to mean+20% by step of 10%	At LV
4-5	Hysteresis	Mean	Mean	Increasing-decreasing-increasing concentration of the pre-calibration levels
4-6	Pressure	Mean	Mean	overpressure 10 mbar and under pressure 5 mbar
4-7	Power supply effect	Mean	Mean	At LV test under 210, 220 and 230 V
4-8	Wind velocity	Mean	Mean	from 1 to 5 m/s (needed?)
4-1	Lab experiments (model)	Mean-10°C, mean, mean -10°C	Mean-20%, mean, mean + 20 %	0, LV, AT for each significant parameters: temperature and humidity (levels) and wind, pressure and interference (2 levels)
4-2	Field experiments			At an automatic station equipped with reference method of measurements
5-1	Cold start, warm start, hot start	Mean	Mean	At LV



4.2 Measurements on site

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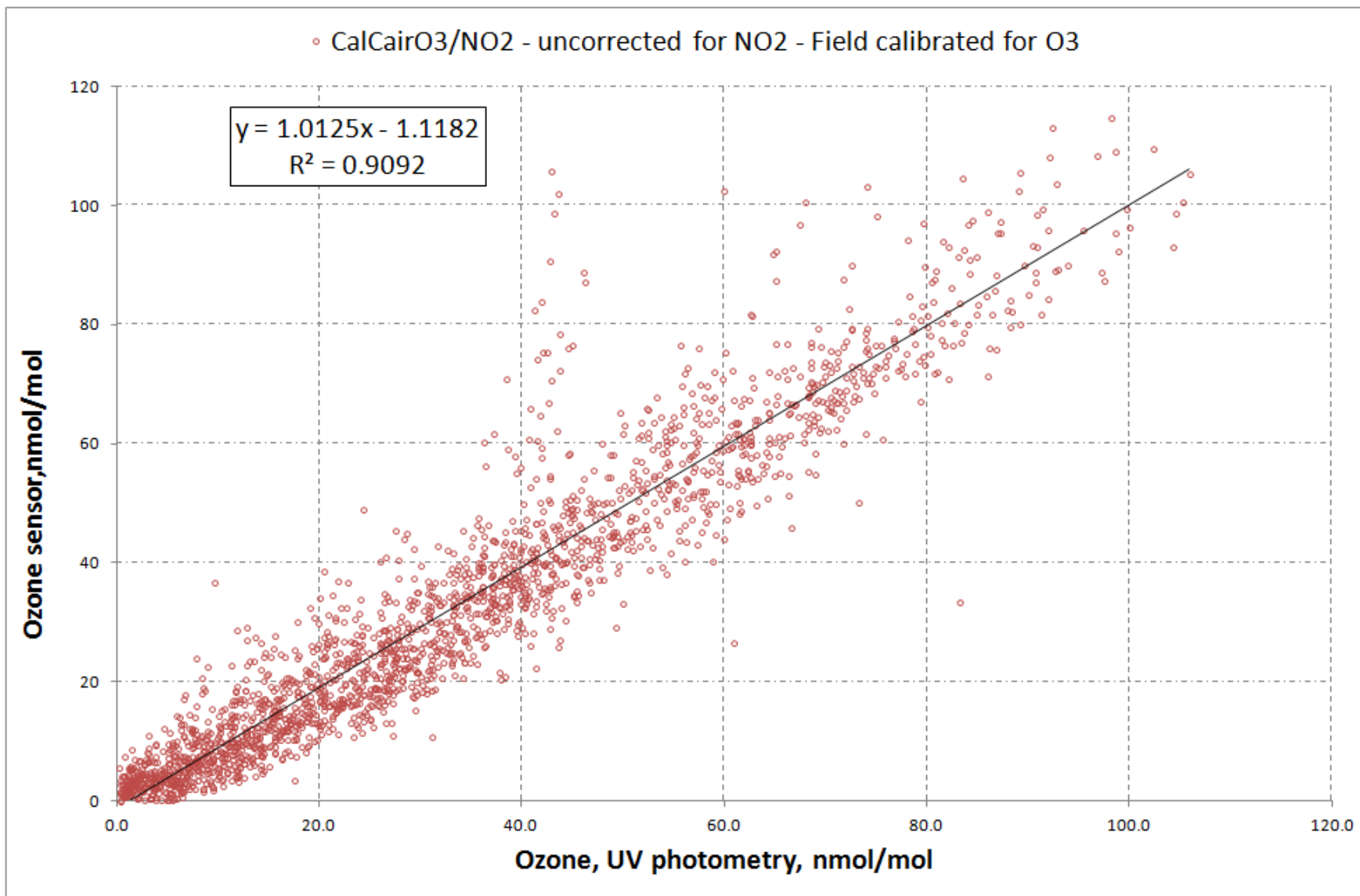


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4.2 Field tests, hourly values





Uncertainty and DQO



$$\blacktriangleright U = 2 (s^2_{\text{lof}} + \cancel{s^2_{\text{bias}}} - s_{r,UV}^2) < 30 \%$$

<i>U, Hourly values, Validation dataset</i>	
60 ppb	26 %
90 ppb	19 %
120 ppb	17 %



Thank you !



O₃ Sensors



Manufacturer	Model	Type
Unitec s.r.l – IT	O ₃ Sens 3000	
Ingenieros Assessores – SP	NanoENvi mote and MicroSAD datalogger, with Oz-47 sensor	
αSense - UK	O ₃ sensors (O3B4)	
Citytech – G	Sensoric 4-20 mA Transmitter Board with O3E1 sensor	
Citytech – G	Sensoric 4-20 mA Transmitter Board with O3E1F sensor	
CairPol – F	CairClip O3	
e2V – CH	MiCS-2610 sensor and OMC2 datalogger,	
e2V – CH	MiCS Oz-47 sensor and OMC3 datalogger	
IMN2P – FR	Prototype WO3 sensor with MICS-EK1 Sensor Evaluation Kit	
FIS - J	SP-61 sensor and evaluation test board	



NO₂ Sensors



Manufacturer	Model
Unitec s.r.l – IT	Sens 3000
Ingenieros Assesores – SP	NanoENvi mote and MicroSAD datalogger, unidentified sensor probably e2v-MICS sensor
αSense – UK	NO ₂ sensors (B4)
Citytech – G	Sensoric 4-20 mA Transmitter Board with 3E50/3E100 sensor
Citytech – UK	A3OZ EnviroceL (for now without test board?)
MIKES – FI	Prototype graphene sensors
InRim – IT	Prototype graphene sensors
CairPol – F	CairClip NO ₂ /O ₃ - filtered

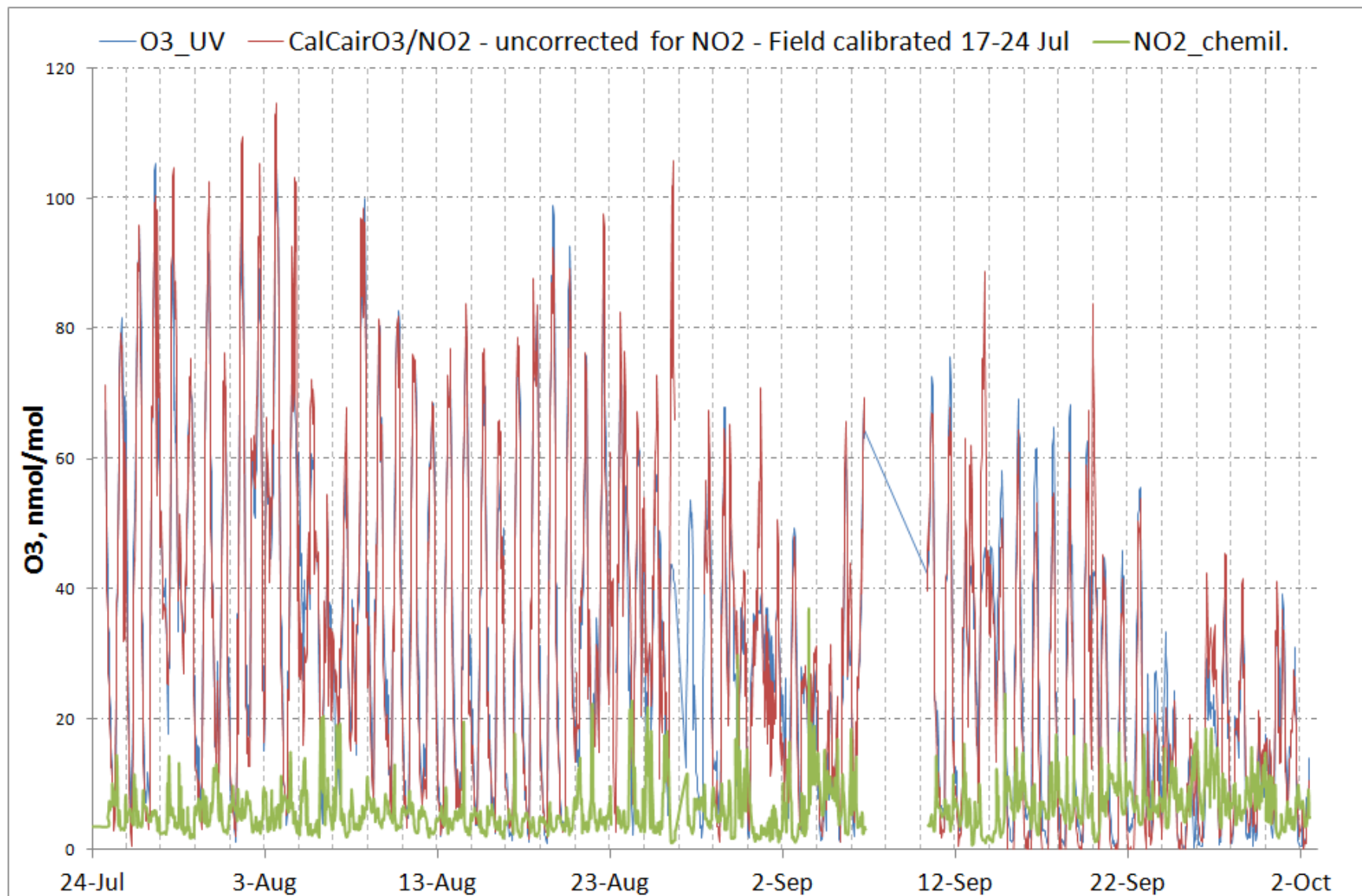


Field tests, hourly values

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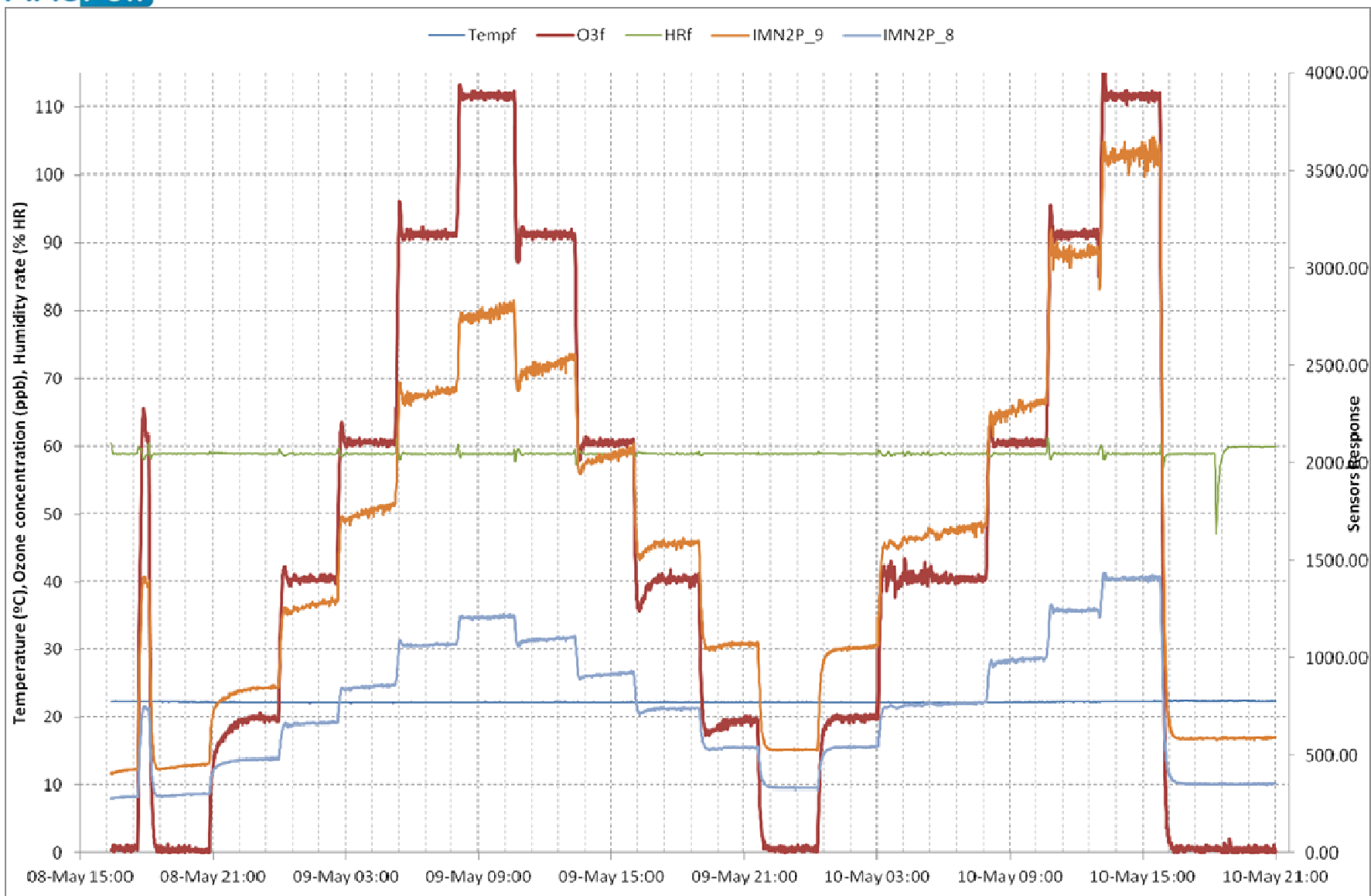


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Hysteresis cycle





4.2 Gaseous Interference

