



Model of a Standard Operating Procedure (SOP) for NO_x measurements with a chemiluminescence analyzer

Aim of this specimen SOP

This model SOP aims at giving support to network operators in setting-up or updating a SOP for NO_x monitoring. It gives example text for the necessary points which need to be addressed in a SOP. However, it can only serve as an extended template because a SOP needs to describe the actual circumstances of a network (site locations, exact type of an analyzer, organization and planning of the maintenance procedures etc.). Thus, these items have to be formulated for each monitoring network individually and in a specific way.

*Example text is written in **normal style**, explanations and directions for needed specific input in **italic style**.*

SOP version

Give date and number of the current version of the SOP.

Responsible person

Name of the author of the SOP.

Implementation of this SOP version

Date of the setting into force of the current version of the SOP and visa of the authorized person.

Measurement principle

Nitrogen oxides (NO_x) are measured continuously by chemiluminescence, after reaction of nitrogen monoxide with ozone. Nitrogen monoxide (NO) is measured directly; nitrogen dioxide (NO₂) is first converted to nitrogen monoxide, after which the sum of the concentrations of both oxides is measured. By assuring that the conversion efficiency is above 98%, the concentration of nitrogen dioxide can then be calculated by subtraction of the independently measured concentration of nitrogen monoxide. The concentrations of nitrogen monoxide and nitrogen dioxide are measured in units of ppbv. For reporting these are converted to units of $\mu\text{g.m}^{-3}$ at standard temperature and pressure (20 °C, 101,325 kPa) using standard conversion factors.

The measurement ranges are 0-1200 $\mu\text{g.m}^{-3}$ for nitrogen monoxide and 0-500 $\mu\text{g.m}^{-3}$ for nitrogen dioxide. Measurement results are fully traceable to internationally accepted standards. The expanded measurement



uncertainties for nitrogen dioxide, referred to the reference periods of the EU air quality limit values, have been calculated in conformity with EN 14211 to be:

- 15 % for a one-hour period
- 15 % for a one-year period, both at the 95% confidence level.

Users of the SOP

Specify here the group of people who will have to work according to the SOP.

Equipment

- Nitrogen oxides monitor (*give type, manufacturer with contact*)
- Sampling line, sampling pump, calibration system (calibration gas, zero air source, dilution system, flow meters etc.) (*give types and short description including manufacturer/provider with contact*)
- Station data logger (*give type, manufacturer with contact*)

General remarks:

The procedures described are in conformity with the relevant clauses of EN 14211 and the manufacturer's recommendations.

The NO_x monitor produces 10-minute-average measurement results for NO_x, NO and NO₂. These results are acquired by the station data processor and digitally transmitted to the central acquisition. In addition, monitor status parameters as listed below are acquired and transmitted.

Give here the list of status parameters.

Installation

Principle:

The analyzer is installed at a monitoring station in a way that allows correct operation, i.e. in a shelter protecting the instrument from dust, rain and snow, direct sun radiation, and with an air conditioning providing sufficient temperature stability to fulfill the requirements of the instrument manual.

Transport:

The analyzer is transferred to the station with attention not to damage internal components. Whenever it is placed in a vehicle care should be taken to protect the instrument from vibrations, direct sun light etc.



Initial Check:

Check to make sure the instrument arrived undamaged. If you find damage, report it in the LOG-book of the instrument. Analyzers are shipped ready to use. Occasionally, however, rough handling during the transport can cause disconnected cables or electronic boards, or incorrectly positioned switches. Verify that the instrument is in operating condition.

A specific procedure for the instrument or some practical hints may be given here.

Rack Installation:

Select a suitable location for the analyzer with sufficient ventilation and convenient access to the front panel display and connections.

The analyzer is supplied with the chassis slides to be mounted in a rack. The instrument requires a properly ventilated rack enclosure in order to avoid operation of the analyzer outside of specifications. The temperature should be in the range of 20 to 30 °C inside the rack enclosure.

After the analyzer has been mounted, make the pneumatic and electrical connections. All pneumatic connections must be tight to ensure accurate operation of the analyzer. Tubing used for sample gas and exhaust connections are 1/4 inch OD and 1/8 to 3/16 inch ID. Only use lines and fittings made of polytetrafluoroethylene (PTFE), perfluoro-ethylene-propylene (FEP), borosilicate glass or stainless steel.

The analyzer is connected to the data logger with an RS232 cable. Ensure that the cable is securely connected to the correct COM port of the logger.

Exhaust Connections:

Connect the exhaust port of the analyzer to the vacuum pump. The pump must be protected with a charcoal scrubber to remove excess ozone and prevent damage to the pump. Connect the exhaust of the pump to an exhaust line.

Setting into operation:

When the instrument is initially powered up, several components in the instrument are required to reach operating temperature before the analyzer will begin operation. This process typically requires about 60 minutes.

Add here a description of the start-up procedure as described in the manual or refer to the manual.

After the instrument has warmed up and has advanced from the start up sequence to the measurement mode perform zero and span check to verify the correct calibration of the instrument. Adjust as necessary.



Routine maintenance

Perform the scheduled maintenance as required by the maintenance schedule. All performed maintenance steps have to be documented in the LOG-book together with date, time and name of the operator.

Here the different maintenance activities should be described. They may comprise e.g.: Flow controls, leak tests, cleaning processes, filter and scrubber replacements, check of sensors and status signals. A full list of required maintenance procedures and schedules can always be found in the instrument manual.

Calibration

Analysers are fully calibrated and tested for linearity (lack of fit, EN 14211)

- Upon receipt (new)
- Every 3 months
- After corrective maintenance.

The calibrations upon receipt and after corrective maintenance are performed in the laboratory, the 3-monthly calibration is performed in the stations.

For this purpose, different gas concentrations are produced from certified concentrated calibration gases using a dynamic dilutor (*specify type*). From the measurement signals the calibration function is determined. The calibration function is used to calculate the NO response factor to be used for further calculations. In addition, a converter efficiency test is performed at concentration levels of 400, 300, 200 and 100 ppb NO₂.

Calculation of results:

Monitor results are expressed in units of ppbv. For reporting, these are converted into units of $\mu\text{g.m}^{-3}$ at standard temperature and pressure (20 °C, 101,3 kPa) as follows:

$$\text{NO } (\mu\text{g.m}^{-3}, \text{STP}) = \text{NO (ppbv)} \times 1,25$$

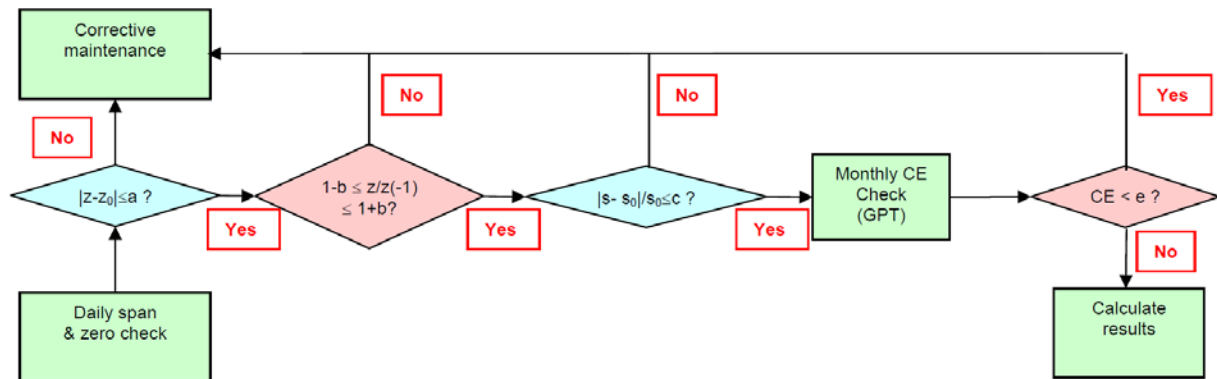
$$\text{NO}_2 (\mu\text{g.m}^{-3}, \text{STP}) = \text{NO}_2 (\text{ppbv}) \times 1,91$$

Quality control

Zero and span checks:

Zero and span checks are performed automatically every 24 hours by the dynamic dilution system.

An example of a decision scheme associated with the results of the checks is given below.



z_0 =zero directly after installation; z =measured zero; $z(-1)$ previous zero

s_0 =span directly after installation; s =measured span

CE=converter efficiency

Possible decision criteria for the decision scheme above are e.g.:

$a=5\text{ppbv}$; $b=0.025$; $c=0.05$; $e=0.95$

Documentation and forms

In the appendix examples of a checklist for general controls in the stations (Appendix 1) and for the documentation of the calibrations (Appendix 2) are given. In addition Appendix 3 shows an example for a detailed description of the calibration procedure.

All completed forms are stored in the LOG book together with the following information:

- name of the operator
- date, time and location
- model and serial number of analyser and calibrator
- gas cylinder serial number and cylinder concentration (observe the cylinder expiry date on the cylinder certificate).



References

EN 14211 (2005). Ambient Air Quality-Standard method for the measurement of the concentration of nitrogen dioxide and nitrogen monoxide by chemiluminescence.

Revision History

In this paragraph the history of revisions of the SOP is documented. It is sufficient to list here the revision dates together with keywords of the revised items. However, all old versions have to be stored at a secure place defined here.

Acknowledgement

Valuable input was provided by the Air Quality Section of the Department of Labour Inspection of Cyprus and by the Swiss National Monitoring Network NABEL.


Appendix 1: Example of a checklist for general controls in the stations:
General checks
Site: Zurich

	fulfilled	
barometric pressure	970.2	Imbar
pump of main sampling line working	yes	
air conditioning working	yes	
water from zero air compressor drained	yes	
pressure at zero air compressor > 4 bar	yes	
temperature in pump compartment around 20°C	yes	
inlet filters changed (only NOx and CO)	yes	
still sufficient filters in stock	yes	
VOC-GC: water in H2-generator filled	yes	
cylinder pressures recorded	yes	
SO2 cylinder	69	bar
NO cylinder	114	bar
CO cylinder	133	bar
CH4 cylinder	138	bar
synthetic air	18	bar

Final control

All MKAL: Mode = Automatic, Stat = Measure	yes
Easycomp: maintenance switched off	yes
PC: backup made, HandyDrive removed from adaptor	
data acquisition started	yes
climatisation working	yes

Remarks:

 Site possibly influenced by nearby construction work from 3 - 6
October 2011



Appendix 2: Example of a Calibration protocol

NO_x-Analyzer Thermo 42i TL

					fulfilled	
Analyzer working ok	(or Alarm:				yes	
Temperatures: Chamber:	48 bis 52 °C				50.4 °C	yes
Cooler:	-12 ± 6 °C				-13.7 °C	yes
Converter:	320 ± 10 °C				325 °C	yes
Pressure:	275 ± 30 mmHg (lowest value)				263 mm Hg	yes
Flow: Sample:	1.2 ± 0.3 l/Min. (lowest value)				1.02 l/min	yes
Ozonator:	Ok?				yes	

Calibration: MKal VM: 134-2.060053

Target value: 81.1 ppb NO/NO_x


		old		new	
Zero :		0.100 ppb NO		0.000 ppb NO	
		0.141 ppb NO _x		0.030 ppb NO _x	
		0.041 ppb NO ₂		0.030 ppb NO ₂	
		old		new	
Span :		75.33 ppb NO	-7.1%	82.00 ppb NO	1.1%
		75.30 ppb NO _x	-7.2%	81.90 ppb NO _x	0.9%
		-0.03 ppb NO ₂		-0.10 ppb NO ₂	
		correction factor NO: 1.077		correction factor NO: 0.989	
		correction factor NO _x : 1.077		correction factor NO _x : 0.991	

Remarks:



Appendix 3: Example for detailed working instructions for calibrations

Input provided by the Air Quality Section of the Department of Labour Inspection (DLI) of Cyprus

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Prepared by:_____ Date:_____


Reviewed by:_____ Date:_____

Approved by:_____ Date:_____

The colored ink stamp indicates this is a controlled document. Absence of color indicates this copy is not controlled and will not receive revision updates.

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Purpose

To describe the procedure to be followed for performing calibrations, linearity tests and converter efficiency tests of the EC9841B NO_x analysers.

Principle

This SOP applies to EC9841B NO_x analysers. The procedures described are in conformity with EN 14211.

Staff involved

This SOP is intended for the staff of DLI and the maintenance company who perform the activities described.

Equipment

- Nitrogen oxides monitor (Ecotech EC9841B)
- Sabio 4010 Dynamic Calibrator
- Zero Air Source (Sabio 1001)
- Certified gas standard of NO in nitrogen
- Station data processor (Ecotech)

Documentation and forms

For calibration and linearity testing the form *DLI-F-3 NO_x Analyser Calibration Report* is required.


The completed form is stored in electronic form in the file *DLI-F-3 NO_x Analyser Calibration Report-sn-yyyy-mm-dd.xls*, where *sn* denotes the analyser serial number, *yyyy* the year, *mm* the month and *dd* the date of the calibration.

For the converter efficiency test, the form *DLI-F-4 NO_x GPT Analyser Calibration Report* is required. The completed form is stored in electronic form in the file *DLI-F-4 NO_x GPT Analyser Calibration Report -sn-yyyy-mm-dd.xls*.

Paper copies of the forms are stored in the history log book of the analyser.

Procedure

General

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Perform the scheduled activities as required by the maintenance schedule (DLI-QD-103).

Registration of general information and status parameters

Registration of general information:


- Enter name of customer, analyser model, analyser serial number and job number in **Customer, Instrument, ID No. and System/Job No.** fields respectively.
- Enter name of operator, date of test, start time and where the test was performed in **Calibration Performed by, Date, Time Begin** (left field) and **Location** fields respectively.

Registration of reference information (section Calibration Equipment, where applicable):

- Enter calibrator model, calibrator serial number, zero air generator model and zero air generator serial number in **Calibrator Model, ID/Serial No., Zero Air Source Model and ID/Serial No.** fields respectively.
- Enter gas cylinder serial number and cylinder concentration in **Gas Std. Cylinder Serial No. and Cylinder concentration** fields.
- Observe the cylinder expiry date on the cylinder certificate and enter it into the **Cylinder Expiry Date** field.
- Observe the cylinder pressure (right gauge) and enter the reading including its unit in the **Cylinder Pressure** field. Remember to read on the Bar scale.

Registration of analyser status parameters (section Displayed Instrument Parameters):

- On the analyser keyboard hit <Exit> to return to the primary screen.
- Push the Select button to enter the main menu. Use the Up and Down arrows on the keyboard to navigate in the main menu. Push <Return> to select a menu item.
- Choose **INSTRUMENT STATUS**.
- From the **INSTRUMENT STATUS** menu read the status parameters and enter them into their respective fields in the **Displayed Instrument Parameters** section of the form.
- Hit <Exit> to return to the primary screen.
- Hit Select to enter the main menu, choose **SYSTEM TEMPERATURES**.
- From the **SYSTEM TEMPERATURES** menu read the status parameters and enter them into their respective fields in the **Displayed Instrument Parameters** section of the form.
- Hit <Exit> to return to the primary screen.
- Hit Select to enter the main menu, choose **TEST MENU**.
- In the **TEST MENU** choose **OUTPUT TEST** menu.
- In the **OUTPUT TEST** menu choose **PREPROCESSOR POTS**.


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- From the **PREPROCESSOR POTS** menu read the status parameters and enter them into their respective fields in the **Displayed Instrument Parameters** section of the form.
- Hit <Exit> to return to the primary screen.
- From the primary screen record the measurement units in the **Instrument units** field.
- Compare the recorded values with the acceptance limits. If any values are outside the limits the cause must be investigated.

Single point calibration

Pre-calibration check (manually):


- Make sure that the analyser reports measurement values in ppb (not µg/m³).
- Press <Exit> to return to the primary screen.
- From the primary screen read the **INSTRUMENT GAIN** and record it in the **Initial Span Instrument Gain** field in the **Single Point Calibration** section of the form.
- Enter the expected measurement range at the particular site in the **Full Scale** field.
- Start calibration from the logger manual/start calibration, NO, “single point all”, select zero point and then press run point.
- Let the analyser measure zero air for 20 minutes and until the reading is stable. The first 10min will be used for the stabilization of the analyzer. For the next 10min we will take 5 measurements (one every two minutes). The average of the five values will be recorded.
- From the Primary Screen, start the calibration sequence by pressing either the **Up** or **Down** arrow key until the display prompts, **START MANUAL CALIBRATION? ZERO**. Confirm that the display reads **ZERO** and press <Enter>. A backlit cursor will be displayed on the NO concentration display.
- Use the <Select> key to move the position of the backlit cursor, and the **Up** and **Down** arrow keys to increment and decrement the value of the backlit digit until the NO zero value is displayed (e.g., 0.000 ppb). When the desired concentration is displayed, press <Enter>.
- Enter the zero air flow rate in the **Precalibration Check – Zero** field of the **Zero flow** column.
- Enter the concentration as measured by the analyser in the **Measured NO** field. Remember to include the unit.
- Start calibration from the logger manual/start calibration, NO, “single point all”, select zero point and then press run point.
- Start calibration from the logger manual/start calibration, NO, “single point all”, select span point and then press run sequence.

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- From the primary screen, start the calibration sequence by pressing either the **Up** or **Down** arrow key until the display prompts **START MANUAL CALIBRATION**. Pressing the <Select> key will allow you to choose from: **NO**, **SPAN** or **ZERO**. Confirm that the display reads **SPAN** and press <Enter>.
- Use the <Select> key to move the position of the backlit cursor, and the **Up** and **Down** arrow keys to increment and decrement the value of the backlit digit until the span calibration gas concentration value is displayed. When the desired concentration is displayed, press <Enter>.
- Let the calibrator generate a concentration of 80% of the NO measurement range.
- Let the analyser measure span gas air for 20 minutes and until the reading is stable. The first 10min will be used for the stabilization of the analyzer. For the next 10min we will take 5 measurements (one every two minutes). The average of the five values will be recorded.
- Enter the zero flow rate in the **80% FS** field of the **Zero flow** column.
- Enter the span gas flow rate in the **80% FS** field of the **Span flow** column.
- The concentration is calculated by the spreadsheet and shown in column **Calculated**.
- Enter the concentration as measured by the analyser in the **Measured NO** field. Remember to include the unit.
- Copy the calculated concentration into the **Expected** field.
- If the error reported in the **Error** column is larger than 2% the analyser gain should be adjusted. If so continue with the next section. If not skip it.

Calibration – Gain adjustment:

- While continuing to measure the span gas press <**Down arrow**> in the primary screen to initiate a calibration of the analyser.
- The display prompts **START MANUAL CALIBRATION? SPAN**. Press <Enter>.
- Enter the span calibration gas concentration value by using the <Select> key to move the position of the backlit cursor, and the <**Up arrow**> and <**Down arrow**> keys to increment and decrement the value of the backlit digit. When the desired concentration is displayed, press <Enter>.
- Move the backlit cursor to the **INSTRUMENT GAIN** field. Press <Enter> to confirm the new value and <**Exit**> to return to the primary screen. The concentration on the *primary screen* should now read the same as the concentration of the calibration gas.
- Enter the zero flow rate in the **Postcalibration Check - 80% FS** field of the **Zero flow** column.
- Enter the span gas flow rate in the **80% FS** field of the **Span flow** column.
- Enter the concentration as measured by the analyser in the **Measured NO** field. Remember to include the unit.
- Copy the calculated concentration into the **Expected** field.

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- If the error reported in the **Error** column still is larger than 2% the problem must be investigated.
- Repeat the zero test and enter the results into the form.
- Record the new instrument gain in the **Final Span Instrument Gain** field.
- If a linearity check will be performed leave the calibrator on and continue with the next section. If not enter **N** in the **Multipoint Linearity Check – Relevant?** field and skip the next section.

Linearity test


- Enter **Y** in the **Multipoint Linearity Check – Relevant?** field.
- Generate concentrations at 80%, 40%, 0%, 60%, 20% and 95% of the measurement range at the site.
- At each concentration at least 4 response time shall be taken into account before the next measurement is performed.
- Enter gas flows, expected concentrations and readings in the **Zero flow**, **Span flow**, **Expected** and **Measured** fields respectively.
- Turn off the generation of calibration gas and switch the analyser back to measuring ambient air.
- The regression line is drawn automatically and some statistics calculated.
- The linearity test fails if the largest relative residual is >5% of its measured value.
- If a converter efficiency test will be performed, leave the calibrator on and return to the **START MANUAL CALIBRATION? SPAN** menu. Confirm that the display reads **SPAN** and press <Enter>.
- Read the converter efficiency and enter it into the **Initial Converter Efficiency** field.
- Compare the recorded values with the acceptance limits. If any values are outside the limits the cause must be investigated.

Converter efficiency test

- The converter efficiency test is performed at concentrations of 95% of the measurement range of NO₂.
- Press <Exit> to return to the primary screen. Press <Up arrow> in the primary screen, press select, Press <Up arrow> twice, press enter. Press <down arrow>, select, Press <Up arrow> change the value to 100%.
- Press <Exit> to return to the primary screen.
- Go to the calibrator screen select **Sequence, run, operator stepper, GPT**, press enter, select point 200ppb NO with zero ozone and press enter.
- Let the calibrator generate a NO concentration of approx. 80% of the NO measurement range.

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- Go to the analyser screen, press enter, **calibration menu**, enter, **calibration mode**, enter, **arrow up** to select **span**, enter, exit
- Let the analyser measure the span gas for at least 20 minutes and until the reading is stable.
- Record the NO, NO₂ and NO_x readings in the first line of columns **NO**, **NO₂** and **NO_x** respectively on the GPT Analyser Calibration Report form.
- While keeping the NO concentration unchanged let the calibrator generate an O₃ concentration equal to 90% of the expected NO₂ measurement range, typically 180 ppb.
- Let the analyser measure the span gas for at least 20 minutes and until the reading is stable.
- Record the NO, NO₂ and NO_x readings in the second line of columns **NO**, **NO₂** and **NO_x** respectively. The converter efficiency is calculated automatically in the spread sheet.
- The converter must have an efficiency better than or equal to 96%. If the converter efficiency is less than 96% the converter should be replaced.
- While keeping the NO concentration unchanged let the calibrator generate an O₃ concentration equal to 60% of the expected NO₂ measurement range, typically 100 ppb.
- Let the analyser measure the span gas for at least 20 minutes and until the reading is stable.
- Record the NO, NO₂ and NO_x readings in the second line of columns **NO**, **NO₂** and **NO_x** respectively. The converter efficiency is calculated automatically in the spread sheet.
- While keeping the NO concentration unchanged let the calibrator generate an O₃ concentration equal to 30% of the expected NO₂ measurement range, typically 50 ppb.
- Let the analyser measure the span gas for at least 20 minutes and until the reading is stable.
- Record the NO, NO₂ and NO_x readings in the second line of columns **NO**, **NO₂** and **NO_x** respectively. The converter efficiency is calculated automatically in the spread sheet.
- The analyser passes the converter test if the converter efficiency is larger than or equal to 96%. If the converter efficiency is less than 96% the converter should be replaced.
- Set the new converter efficiency on the analyser.
- Stop the calibrator by pressing end sequence until exit.
- Go to the analyser screen, press enter, **calibration menu**, enter, **calibration mode**, enter, **arrow up** to select **measure**, enter, exit

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Finalizing the visit and completing the form

- Enter the end time of the calibration in the **Time Begin/End** field (right field) in the form.
- Sign the form in the **Technicians Signature** and **Date** fields.
- Before leaving the station record the visit in the station visit log.
- After returning to the lab store the form in the instrument history log book.

Relevant documentation

Maintenance schedule

Form *DLI-F-3 NOx Analyser Calibration Report*

Form *DLI-F-4 NOx Analyser GPT Calibration Report*.

Reference Procedures

Nitrogen oxides monitor (Ecotech EC9841B) manual

Sabio 4010 Dynamic Calibrator manual.

Revision History

Revision 0