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Preparations for PAN instrument intercomparison

Offenbach, NILUs first visit 7 to 9 September 1993

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1. Introduction

PAN (peroxyacetyl nitrate) is a component of photo-chemical smog and an important element in the total budget of organic nitrogen compounds in the atmosphere. PAN is unstable, and therefore complicated to measure. Also the procedures for calibration of PAN monitors are complicated. An ongoing STEP project (EV4V-CT90-0222) addresses the calibration of PAN-standards in liquid solution. Also other elements are essential in the quality control of PAN measurements. The two most important elements are the calibration of the PAN instrument and the response of the instrument to the ambient air.

The Umweltbundesamt (UBA) in Offenbach (close to Frankfurt am Main) has therefore initiated a PAN instrument intercomparison. Different instruments are moved to a ring-test laboratory. At this site they will be calibrated with different methods and will monitor ambient air for a period of approximately 4 weeks. The calibration methods, calibration results and monitoring results will be compared, and any discrepancies, non-linearities etc be discussed in a paper.

2. NILU's expectations

NILU has recently been accredited in selected fields of air sampling, field measurements and chemical analysis (according to EN45001). Our methods for PAN measurements have been included in the accreditation. Due to lack of comparative information, the uncertainties had to be estimated and were set in the range of ±30%. We hope to gain more specific knowledge of the uncertainties in our methods during the intercomparison campaign in Offenbach. We also foresee that similar campaigns with a larger number of participants may be needed in the future.

NILU expects to be able to compare the bag calibration method (NILU's method description is presented as an enclosure in this report) with calibration based on an on-line PAN generator, and with the calibration techniques employed by the other participants.

NILU expects to be able to measure the linearity of the NILU PANalyzer over three decades of PAN concentration (~1 µg PAN/m^3, ~10 µg PAN/m^3, ~100 µg PAN/m^3). We are planning to do this by our own bag calibration method, and to verify the results with other methods available during the intercomparison.

NILU expects to be able to compare the response of the NILU PANalyzer with the response of other PAN instruments when they monitor ambient air in the same location for a period of approximately 4 weeks.

With help of the gas mixing facilities provided in the Offenbach ring-laboratory, NILU also hopes that it may be possible to identify unknown peaks and potentially interfering compounds.

3. Short description of the NILU PANalyzer

The NILU PANalyzer is a gas chromatograph equipped with a simple ECD (electron capture detector. The column is a glass tube, 1 m by 2 mm inner diameter, packed with 0,15% diglycerol and 6% QF-1 on 100-120 mesh Cromosorb G-HP. The column is operated isothermally at 32,5°C. The detector is operated at the same temperature as the column, or slightly elevated (approximately 45°C). The carrier gas is nitrogen grade 5.0, humidified by passing it through a steel cylinder with salt crystals containing crystal water ($MgSO_4 \bullet 7H_2O$). The pressure in the salt cylinder is approximately 9 atmospheres, and the resulting humidity of the carrier gas is in the range 5-10%RH, measured at the outlet. The carrier gas flow is 20-25 ml/min.

The ECD is normally operated in the CF (constant frequency) mode for maximum sensitivity. With 20 V pulses of ~0,7 µs duration at 3,5 kHz, a standing current of 1 nA is achieved. The sample is pumped through a 2 ml sample loop and introduced in the system by a 6-port Valco sample valve. The PAN detection limit with this configuration may be in the range 5-50 ppt, depending on the level of contamination and temperature stability of the system. The level of contamination in the calibration bag normally corresponds to a PAN peak of 25 ppt or lower.

The instrument is automated with a PLS-system, and performs 2-4 samples per hour. The chromatograms are registered and integrated by a Hewlett Packard 3396 integrator. A BASIC program in the integrator automatically sends a one-line report to a PC logger after each sample. The PC logger may use PCPLUS or a custom made program to log the incoming records in an ASCII file, which may later be processed by normal spreadsheet programs.

4. Results from NILU's visit in Offenbach 07-09.09.93

A PAN-instrument should normally be allowed to settle down in normal operation for a period of 1-3 weeks before important measurements. A NILU PANalyzer was installed in the ring-laboratory on 08.09.93. Calibrations the following days revealed that the instrument did not function satisfactorily. Two problems were isolated.

The humidification cylinder was positioned after the second stage pressure regulator, immediately before the sampling valve. In this position the pressure is in the range 0,7-1,2 atmospheres, and the vapour pressure of the water over the salt crystals results in a humidity of ~80%RH. This saturates the detector with water, and the sensitivity is very much reduced.

The support of the packing material was Chromosorb W-HP. This support is more porous than the G-HP. The specific weight of the G-support is approximately twice that of the W-support. Consequently, the separation is much lower for the G-support.

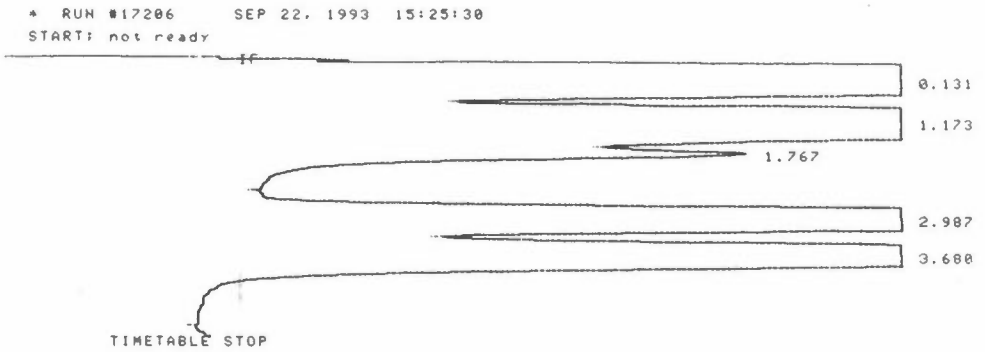
With the W-support, the very reactive PAN is very easily lost due to adsorption and decomposition. We expected that this problem had been solved with the introduction of carrier gas humidification. The column performed very well for some days. After installation in Offenbach the response to PAN was decreased by a factor of 10 compared to the stable internal standard NPN (n-propyl nitrate). The conclusion is that the W-support is too fragile to withstand the pressure transients during sample valve switching. Some particles are crushed, and unsilanized surfaces are exposed. This may happen already during careless packing of the column, during transport, or during normal operation of the instrument. The column in the instrument first installed in Offenbach has Chromosorb W-HP support, and will be replaced with a Chromosorb G-HP column.

In other respects the system was successfully installed in the ring-laboratory. An outdoor sample intake was constructed with a glass funnel connected to a 1/4" PTFE tube passed out through a hole in the wall. The 1/8" sample intake tube is passed out through the larger tube. The sampling tube may be pulled in to be connected directly to a tedlar bag with a calibration mixture. It may also be connected directly to the glass tube of the ring-laboratory gas-mixing system. If any adsorption or contamination should occur in the intake tube, the effect will be exactly the same regardless of which system the intake tube is connected to. This provides an excellent basis for comparison of different methods and gas mixtures.

5. Further preparations

The intercomparison experiments are scheduled to start on a Monday. The Friday before, NILU's representative will rebuild the instrument in the Offenbach ring-laboratory with a new (pre-conditioned) column. The humidification cylinder will be connected at a higher pressure to reduce the carrier gas humidity. The instrument will then be allowed to settle down during the week-end before the intercomparison experiments are started. A longer settling period would be preferred, but does not seem to be so important when the carrier gas is slightly humid.

In case of unexpected problems, NILU will also bring a second PANalyzer to the intercomparison experiments. The best of the two instruments will be left in Offenbach for the ambient monitoring experiment.



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3.680	21556976	SPB	.314	5.71240

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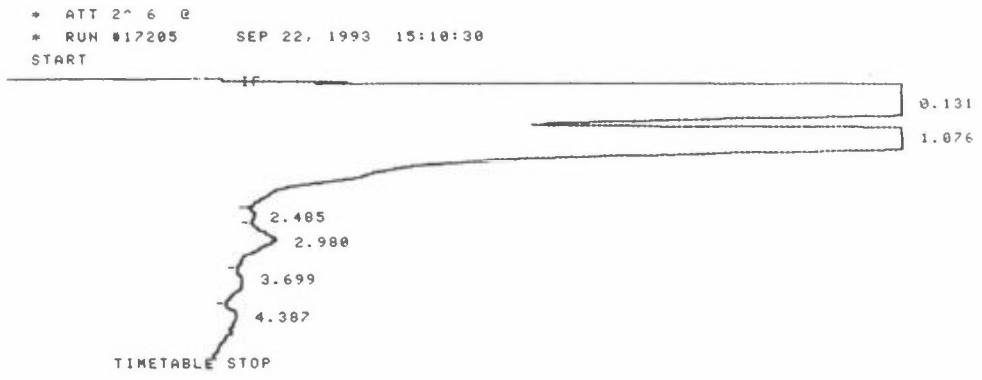
MUL FACTOR=1.0000E+00

Figure 1: A calibration sample containing approximately 12 µg PAN/m³ (retention time 2,987 minutes) and 10 µg NPN/m³ (retention time 3,680 minutes). A concentration of 1 ppt of PAN would give a peak area of 19000, 1 ppt of NPN corresponds to an area of approximately 14000. This sample is made with NILU PANalyzer number 2 (normally stationed in Southern Norway) in the NILU laboratory. The column packing material is 0,15% diglycerol + 6% QF-1 on Chromosorb G-HP 100-120 mesh.



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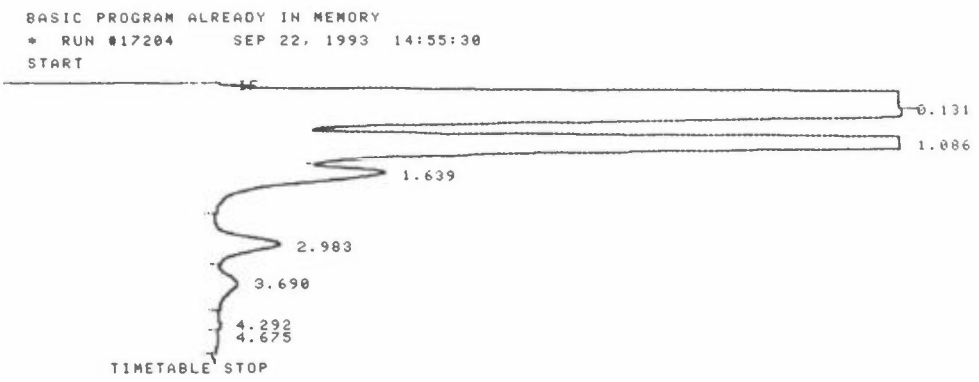
MUL FACTOR=1.0000E+00

Figure 2: The blank bag has a "memory" or contamination that corresponds to approximately 22 ppt of PAN. The integrator plot attenuation is set to 6.



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3.690	1034636	BB	.350	.54624
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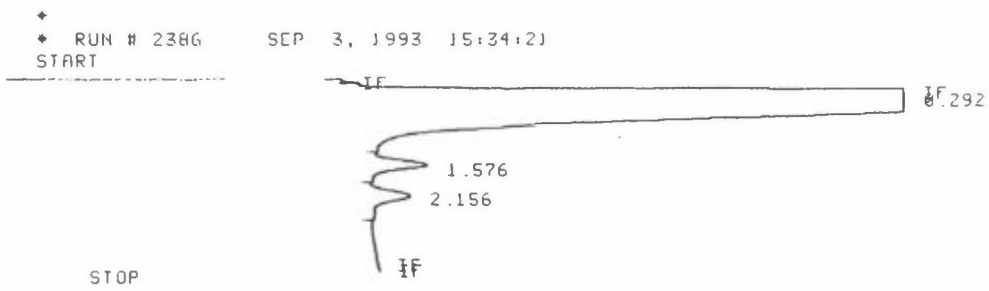
MUL FACTOR=1.0000E+00

Figure 3: A sample of air from the NILU laboratory at integrator attenuation 8 (the scale is reduced with a factor 4 compared to the previous figure). The PAN peak at retention time 2,983 minutes corresponds to a concentration of approximately 152 ppt. The peak at 3,690 minutes may be PPN (peroxypropyonyl nitrate), but it has not been identified or calibrated in this instrument type. The peak at 1,086 minutes is water, the peak at 1,639 minutes has not been identified.

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DIRECTORY FULL

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MUL FACTOR=1.0000E+00
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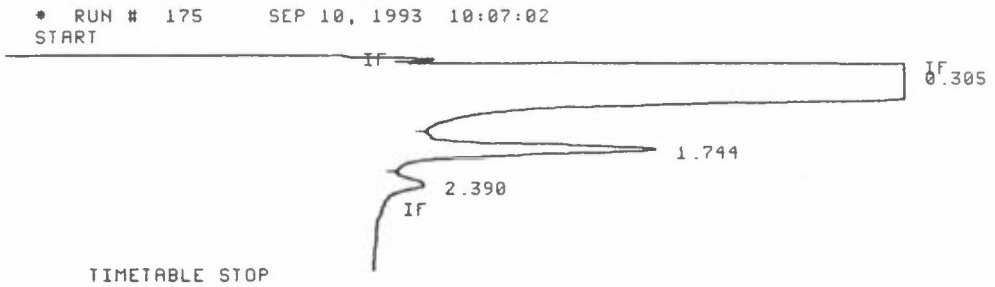
Figure 4: Calibration sample from NILU PANalyzer number 6 (normally stationed at Spitsbergen) made in the NILU laboratory before transport to Offenbach. The column packing material is 5% CW400 on Chromosorb W-HP 100-120 mesh. The sensitivity is relatively low due to excessive carrier gas moistening. Note the excellent separation between PAN (at retention time 2,156 minutes) and NPN (at retention time 1,576 minutes). The ratio of the PAN and NPN response factors is approximately 0,7, a quite normal result with the Chromosorb W-HP support.



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MUL FACTOR=1.0000E+00

Figure 5: After installation at Offenbach a calibration similar to that of Fig. 4 reveals that the PAN response factor has decreased very much compared to the NPN response factor. This indicates that the column support is becoming more reactive in spite of the moist carrier gas. The column must be replaced before further experiments are performed with this instrument. The more stable Chromosorb G-HP must be preferred in spite of the reduced resolution.

Appendix 1

Tedlar bag calibration of PAN GC

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Tedlar bag calibration of PAN GC

1. Introduction

When a solution of PAN (peroxyacetyl nitrate) with known concentration is given, it may be used for calibrating an ambient air PAN monitor. The present method has been the standard method at NILU since 1986, only small changes have been made during the period until october 1993. In the october 1993 version humidity is added to the bag to deactivate the internal surfaces. This significantly improves the accuracy of the method.

2. Theoretical background

The method falls into the category static dilutions of PAN. A very small amount of PAN dissolved in a solvent (pentane or hexane) is injected into a tedlar bag using a glass syringe, and quickly evaporates. The initial PAN concentration in the bag is then quite well known (except for eventual adsorbtion loss in the syringe and evaporation loss during opening of the standard bottle). Some PAN will be lost in adsorbtion at the walls of the bag. However, water is added to the bag first, and all reactive surface spots are covered with water vapour. Since PAN is not easily dissolved in water, the walls are very inert, and the adsorption loss of PAN is expected to be small.

The PAN concentration will decay exponentially with time due to thermal breakdown of PAN. This decay is measured by registering the concentration in the bag in 4-8 consecutive samples. The thermal decay may be plotted and the concentration extrapolated back to the time when the standard was injected into the bag.

The instrument dynamic range and linearity is not checked in a normal calibration. These parameteres are not expected to change much as long as the sensitivity is stable or decreases slowly with time. A normal calibration consists of a minimum of 1 blank and 4 calibration samples from a bag with low PAN concentration (approximately 5 to 50 times the detection limit) and 1 blank and 4 samples from a bag with high PAN concentration (approximately 5 to 50 times the low concentration). The instrument operates in its normal automatic cycle. The intake tube is pulled in through the wall and connected to the calibration sample bags. In this way the whole intake system is included in the measured instrument sensitivity.

3. Chemicals

- PAN standard (liquid solution)
- Clean, pressurized air (synthetic air) from bottle or generator
- Methanol and hexane (residue analysis grade) for equipment cleaning

- Distilled, filtered water (milli-Q)

4. Equipment

- Tedlar bag with septum (normally 10 l volume) (aluminum foil "sandwich" bags are not usable due to contamination)
- Syringe 10 µl for PAN solution
- Syringe 100-250 µl for water
- Air generator, Nitrox ECA 3000 or similar (not catalytic type) or synthetic air bottle (preferrably HC-free grade)
- Pressure regulator for air bottle (8 bar) or pressure regulator for air generator (1-4 bar). Some air generators have an internal pressure regulator of sufficient stability.
- Restriction (hammered-flat tube or frit)
- Gas flow meter
- SS tubing, PTFE tubing, silicone rubber tubing, fittings

5. Procedure

5.1. Initial preparation of equipment

When the syringes are new, or a contamination is suspected, clean the syringes as follows. Remove the plunger from the syringes and clean them with hexane in a test tube in an ultrasonic bath for some minutes. Repeat with clean solvent. Repeat the same operation with the barrels and needles. Pump approximately 50 ml hexane through each barrel/needle by connection to a suitable vacuum pump with solvent collector.

The pressure regulator may be used without initial cleaning if it is delivered from the factory "cleaned for oxygen service". Make sure that also pressure gauges connected to the regulator are cleaned to the same specification. A metal membrane pressure regulator (all metal, no lubricant, a PTFE valve seat is acceptable) may be cleaned by flushing a solvent through it.

Air Liquide HBS300 regulators employ lubricated O-rings, but have still been used successfully without initial cleaning. If problems are encountered, these regulators may be disassembled, cleaned with methanol in ultrasonics (4-6 times 10 minutes) and dried in vacuum. To clean the manometer, methanol may be pumped through a thin PTFE tube inserted as far as possible into the bourdon tube.

Silicone rubber tubing is used without initial cleaning. Do not use old tubing that may have been contaminated by previous use.

If bottled air is used, prepare a restriction that will allow a flow of approximately 5 l/min with the output pressure of the selected pressure regulator. Clean the restriction and any additional SS tubing by pumping at least 50 ml of hexane through it. Dry the restriction by pumping normal air through with a water suction pump for an hour. Connect the pressure regulator to the bottle and the restriction at the outlet of the regulator. Connect a digital mass-flow meter after the restriction, and a piece of PTFE tubing with a piece of silicon rubber tubing for connection to the tedlar bag. Flush the system at least one hour with hydro carbon free air before using it the first time.

If the Nitrox clean air generator is used, make the restriction to admit approximately 3 l of air at the output pressure of the selected pressure regulator. Connect the system as described above. Start the air generator and flush the system with 3 l/min for approximately one hour. Reduse the pressure so that the flow is approximately 100 ml/min and let the system be flushed overnight. Let the generator operate continuously with the outlet closed, even if it is only used infrequently.

Mark a tedlar bag with "PAN LOW, date initiated" and one with "PAN HIGH, date initiated". An additional station name or initials may identify the place/person that uses these particular bags. Mark two 10 µl syringes in the same manner. Use the "LOW" equipment for the concentration range 1 to 10 µg PAN/ml solvent. Use the "HIGH" equipment for the concentration range 20 to 200 µg PAN/ml solvent.

The flow meters and pressure regulator may contaminate the tubing, the tedlar bag and the PAN instrument. If contamination is suspected, test the equipment in the lab on an instrument that is not employed in regular monitoring.

5.2. Preparation of equipment and check list

Use a photo copy of the check list given at the end of this document. Register the equipment and chemicals used, and all events as indicated in the check list. The completed check list and the figures referenced in the check list are the documentation of the instrument calibration.

If the standard has been transported to the station packed in dry ice (-72°C), put the standard in a normal freezer to heat it to approximately -25°C to -20°C before the bottle is opened.

If a pressure swing clean air generator is used, flush the gas flow measurement system with 100 to 200 ml/min for 30 minutes before use. If an air bottle is used, flush the system at full flow approximately 2 minutes before use.

Connect a flow meter in the sample intake line and measure the sample intake flow of the instrument to be calibrated. This flow multiplied with the sample pumping time is the amount of air pumped through the instrument for one sample.

5.3. Filling the bag with air

Flush the bag once in the following way: Fill it with 5 to 10 l of air, unscrew the septum cap, let the bag empty itself on a flat table top under gentle pressure of a suitable flat object. The bag is easily destroyed if over-pressurized. Replace the septum cap and make sure the inlet valve is open.

Measure the air flow from the bottle or generator. Compute the time required to fill the bag with 10 l + the volume required for one sample. When one blank sample has been drawn from the bag, the bag will contain 10 l of air.

Using a stop watch, connect the bag to the air outlet from the flow meter, and disconnect it after the time computed above. Do not close the bag. If an air bottle is used, close the bottle valve. If an air generator is used, reduce the flow to approximately 100 ml/min.

5.4. Humidification

Inject 100 µl of milli-Q water into the bag and shake the bag well to disperse the droplets. A syringe with a very small opening in the needle will prduce a fine mist of small droplets. The air in the bag will now have approximately 60%RH, and the bag walls will be deactivated after some minutes.

5.5. Blank sample

Pull the instrument inlet tube in through the wall or disconnect it from the central high volume air intake. Connect the tube to the sample bag with a small piece of silicone rubber tubing. Do not stop the automatic sampling of the PAN instrument. Wait for the instrument to sample the bag in its normal automatic cycle.

The blank sample should not show any significant interference in the retention time area of PAN or NPN. Include a copy of the chromatogram with the documentation. If the blank is not acceptable, empty the bag, flush it twice, refill the bag (10l + one sample), add water and repeat the blank sample.

If the blank sample is still not acceptable, repeat the paragraph above. If this does not help, discard the bag and the silicone rubber pieces. If the same interference appears with a new bag, either the air system or the instrument sampling loop is contaminated.

5.6. Standard injection

The PAN standard must be injected into the bag 3 to 10 minutes before the instrument starts the sample pump. The solvent needs some time to evaporate and release the PAN, and the concentration in the bag must reach an equilibrium by diffusion.

Remove the standard bottle from the freezer (using gloves to avoid heating the bottle), open it, and place it on the table. Fill the syringe three times and discard the contents on the floor (this is to avoid cross contamination between standards, some µl of hexane on the floor have no consequence). To remove air bubbles in the syringe, pump the plunger quickly down and slowly up a few times with the needle in the solution. Fill the syringe with a larger volume than required, point the needle upwards and adjust the plunger to the exact volume required (normally 10µl). Insert the needle into the bag through the septum, quickly press the plunger, tap the septum holder sharply to shake off the last drop from the needle, then withdraw the syringe and replace it in its box. Quickly cap the bottle and put it in the freezer. The process should be performed quickly, so that the bottle is not left open on the desk more than 20-30 seconds.

5.7. Calibration samples

The instrument operates continuously in its normal, automated cycle. It is allowed to sample a minimum of 4 samples from the bag. If the PAN response is increasing from sample to sample, adsorbtion in the bag is indicated (caused by too dry surfaces). Normally, the PAN concentration in the bag should fall exponentially with time (due to thermal decomposition), and this should be clearly seen in the calibration samples.

The integrator plot scale should be set to give an enlarged view of the baseline, so that peaks down to the detection limit are clearly visible. Furthermore, peak markers should be enabled, so that the integrator's interpretation of the beginning and end of a peak is documented.

6. Calculations

6.1. Evaluation of blank

A baseline irregularity or interfering peak in the blank is accepted if it is less than 5% of the expected response to the low standard, or less than the practical detection limit.

NILU uses a practical definition of the detection limit. When only peak height and baseline noise are considered, large error sources are ignored, since the integrator does not recognise all peaks correctly. At the practical detection limit, at least 50% of the PAN peaks should be correctly detected and integrated. Reported area should be within ±20% of the true area (when compared to similar peaks with correct identification of peak start and stop). Peaks that are not correctly detected should ideally be reported as missing. Not more than 10% of the PAN peaks at the detection limit should be reported with area outside ±50% of the true value. (Due to PAN adsorption in the calibration equipment, it may be difficult to perform accurate calibration experiments at concentrations below 100 ppt.)

6.2. Evaluation of calibration samples

The PAN concentration in the bag will fall exponentially with time due to normal thermal decomposition of PAN. Plot the registered response to 4 or more consecutive samples from the calibration bag. Extrapolate the response back to the time when the standard was injected into the bag. This intercept represents the instrument sensitivity. When at least 3 samples give a consistent picture, one ore more outliers may be ignored.

The NPN concentration (n-propyl nitrate is normally added as an internal standard in the PAN solutions) should be constant in the bag, and the registered response should be constant.

7. Error sources and accuracy

7.1. Contamination

If a bottle of compressed, synthetic air is used, select a clean grade normally designated "Hydro carbon free". Normal "Synthetic air" may be used. This should be filtered through a stainless steel cartridge with activated carbon. The filter cartridge itself may introduce contamination. The best solution is therefore hydro carbon free synthetic air without additional filtering. Some residual hydrocarbons are not dangerous for the PAN instrument, but the HC-free air bottles and filling equipment are subject to more stringent control by the gas manufacturer.

An even better solution is a clean air generator of the pressure swing type (Nitrox ECA 3000 or similar). This air is filtered in a large molecular sieve bed, which is automatically regenerated.

Never use a clean air generator of the catalytic type. Such instruments do remove hydro carbons etc., but they produce reactive residues that cover the internal surfaces of the PAN instrument and may cause extreme adsorbtion problems.

If a pressure regulator is used for the calibration air, select a high quality type with stainless steel membrane. Rubber membrane pressure regulators will contaminate the PAN instrument.

After solvent cleaning the syringe is not likely to contaminate the PAN instrument. Cross contamination between different PAN standards is eliminated by using two separate syringes for low concentration (1 to 10 µg PAN/ml) and high concentration (20 to 200 µg PAN/ml) standards.

The tedlar bag is used without any initial cleaning. Separate bags are used for low and high concentration standards. Any contamination in the bag will be seen in the blank sample before each calibration. No significant peaks should be found in the retention range of PAN and NPN in the blank sample. Also cross contamination between calibration samples is avoided by examining the blank before proceeding with a standard injection.

Contamination could alter the performance of the PAN instrument, but does normally not introduce calibration errors.

7.2. Adsorbtion

The interior walls of the syringe and the tedlar bag ar not perfectly inert. An unknown amount of PAN is adsorbed on the walls. Presently, this error is estimated to be smaller than 5%. The sensitivity of ion chromatography is too low to allow direct measurement of the PAN recovered by bubbling hydrolysis from a 10 litre bag after injection of 10 µl of a normal PAN standard. Such control experiments may become possible at a later time.

Differences in humidity between normal ambient samples and calibration samples may cause changes in the PAN adsorbtion on the separation column. Such errors are estimated to be small.

7.3. Volumetric errors

The PAN standard is stored at -20°C to -25°C. It is not warmed to room temperature before injection. The actual temperature, and the specific volume, of the standard in the 10 µl syringe is unknown. The syringe could be stored in the freezer before injection to reduce temperature differences. This however increases the possibility of plunger leaks or syringe splitting. The volume of standard injected into the bag is estimated to be within ±5% of the nominal value.

The volume of dry air filled in the bag is measured by a mass flow meter and a stop-watch. The resulting error is within ±5%. Variations in atmospheric pressure, laboratory temperature and ambient air humidity are ignored.

7.4. Integration errors

Individual variations of ±20% in the registered responses are considered normal at or close to the detection limit. Concentrations more than 10 times the detection limit should be registered with fluctuations within ±10% of the expected response.

Systematic errors in the evaluation of peak area in the electronic integrator will affect both the calibration and the ambient air samples. The measurement error is in this case cancelled by a similar calibration error. Non-systematic integration errors may be found during the evaluation of the calibration chromatograms. Such samples are ignored, and the calibration is re-run if the number of acceptable samples is too low. Consequently, integration errors do not contribute to the calibration error budget.

8. References

Station:
Instrument:
Date:
Operator:

Bag identity:
Source of clean air:
Pressure regulator:
Flow meter and restriction:
Instrument sample pumping time and flow:
Resulting sample volume
Required air volume (10 l + volume of 1 sample):
Air source flow and resulting bag filling time:
Water bottle filling date:
Water syringe identity:
Amount of water injected into bag:
Blank accepted after how many flushings?
Copies of chromatograms from all (rejected and accepted) blanks are enclosed with check list:

PAN standard identity:
Concentration of PAN in standard:
Concentration of NPN in standard:
PAN syringe identity:
Volume of standard injected:
Time of injection into bag:
Nominal concentration of PAN in the bag:
Nominal concentration of NPN in the bag:
Number of samples registered:
Copies of all chromatograms enclosed with the check list:
Plot of responses enclosed with the check list:

Computed sesitivity for PAN:
Computed sensitivity for NPN:

Comments:

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Norsk institutt for luftforskning (NILU)
P.O. Box 64, N-2001 Lillestrøm - Norway

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CLIENT
NILU

ABSTRACT
An intercomparison of PAN (peroxyacetyl nitrate) monitors is in progress in the Offenbach branch of the Umweltbundesamt. The report describes recent results obtained with the NILU PANalyzer and the bag calibration method that NILU will employ in the intercomparison.

NORWEGIAN TITLE

DESCRIPTORS		
PAN	instrument intercomparison	tedlar bag calibration

ABSTRACT (in Norwegian)

* *Classification*
- *A Unclassified (can be ordered from NILU)*
- *B Restricted distribution*
- *C Classified (not to be distributed)*