**Deliverable D3.3:**
Development of a measurement standardization and data submission protocol for aerosol particle mass spectroscopy based on Aerosol Chemical Speciation Monitor (ACSM) products

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| Comments | The submission of this deliverable, initially due the end of May 2016, is delayed due to a combination of both logistics and the need to finalize results obtained from the recent ACSM instrument inter-laboratory comparison (ILC).

The first ACSM ILC within ACTRIS 2 was held in February and March 2016. This involved 15 different research groups working together at the Aerosol chemical monitor calibration centre (ACMCC) in Paris. This ILC exercise was divided into two different phases to accommodate the large number of instruments and to characterize the newer model of the ACSM instruments that have recently joined the ACTRIS2 network. The analysis of the data from the ILC and the subsequent production of the instrument reports coincided with the due date for this WP deliverable. This together with the change in the organization of the personnel involved in this work package required more time than initially estimated to prepare this report. |
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1.0 Background and objectives of Task 3.3

This deliverable deals with Task 3.3 of WP3 (“Development of a measurement standardization and data submission protocol for aerosol particle mass spectrometry based on aerosol chemical speciation monitor (ACSM) products”). The aims of this subtask were to implement a set of common European Standard Operating Procedures (SOP) for sampling and analysis of ACSM instruments, in order to facilitate their subsequent implementation at several ACTRIS sites. This task is a continuation of that performed within the EU FP7 Infrastructure Project ACTRIS on ACSM suitability for long-term measurements (WP21, D21.6).

As part of ACTRIS 1, the suitability of the ACSM for long-term measurements was assessed at a number of different ACTRIS sites (e.g. Mace Head, Cabauw, Melpitz, Hyytiala, Finokalia, and Jungfraujoch (Frohlich et al., 2014)). They were shown to be successful in monitoring near real-time and on a yearly basis the chemical composition of the fine aerosols and sources of organic aerosols. Following this, the objectives of these instruments within the ACTRIS-2 Grant Agreement, are:

- to provide homogeneous quality-controlled ACSM datasets at a European scale,
- to homogenize calibration and data processing and in particular the source apportionment of organic aerosol data.
- to assure the precision and accuracy of these measurements through comparison with various co-located instruments e.g TEOM-FDMS, SMPS, OC-EC etc.

This will be achieved via:

- Inter-laboratory comparison (ILC) of instruments and group calibration exercises,
- Regular reporting of instrument calibration performance.
- Homogeneous sampling set ups and analysis tools.
- Implementation of Standard operating procedures.

In addition to the ILC of online aerosol chemistry ACSM instruments, measurements of external tracers from complimentary sampling methods have been performed to determine the precision and accuracy of the ACSM instruments with respect to other techniques. During the ILC, both synthetic standards and ambient air sampling were used to determine the quality performance of the participating laboratories. These exercises, to which each participating site in ACTRIS should attend at least once, ensure that all ACSM instruments within the ACTRIS network are providing quality-controlled data. Attending the ILC guarantees homogenize calibration of these instruments on a European scale.
2.0 First interlaboratory comparison: Description and outcomes

2.1 Description

During the EU FP7 ACTRIS, the first ILC intercomparison of all ACSM instruments to different inorganic mixtures and ambient aerosol samples was examined. These ACSM instruments showed good agreement, which is owed to the identical instrument design and data analysing software that exists within this community (Crenn et al., 2015). However, some differences in the organic mass spectral response was observed between instruments. Using the organic mass spectral data obtained during this intercomparison, a comparative source apportionment (SA) exercise of organic mass spectra from all instruments was performed (Frohlich et al., 2014). Slight differences in the organic signal was observed, but did not influence the source apportionment of different organic factors (or sources) identified by all instruments (Frohlich et al., 2014). As a result of the ACTRIS1, ILC, a number of subsequent laboratory studies were performed to characterize and understand how these ACSM instruments respond to different mixtures of organic and inorganic species.

The instrument uncertainties brought to light after the first intercomparison (variable instrument response to organic aerosol compounds) were one of the main research focuses of the second intercomparison effort (the first ACTRIS 2 ILC). The first ACTRIS 2 ILC took place during February and March 2016, there was 16 different instruments that participated and more than 20 different researchers from 11 different countries (divided over two separate intercomparison periods). The preparation for the ILC took place between September 2015 and January 2016, with the different tasks being distributed among the involved parties. These preparations involved identifying the different calibration procedures and partners to be involved. As well as instructions for laboratory set-up, instrument handling, and reporting.

During the ACTRIS2, ILC, a potential aerosol mass chamber (PAM, Aerodyne ®) was used to generate controlled quantities of secondary organic aerosol (SOA) in order to better characterise the ACSM response to organic aerosol under controlled conditions. This PAM chamber was necessary in order to form realistic samples of SOA. There is currently no other means of doing this from solutions of organic aerosols. The results of these intercomparisons and the analysis of the organic aerosol laboratory studies is currently underway by the Aerosol Chemical Monitor Calibration Centre (ACMCC), with the aims to present results at the upcoming European Aerosol Conference in September 2016.

During the ACTRIS-2 ILC, all instruments sampled under the same conditions (inlet set up, sampling lines etc). Good agreement with the ACSM instruments was observed with collocated external tracers operating along side the ACSM instruments at the SIRTA station, including total submicron particulate matter (PM1), inorganic ions nitrate and sulphate measured by the particle into liquid sampler (Figure 2.0). As part of the ACTRIS-2 ILC, two separate workshops were organised. The first focused on instrument improvements and was lead by the...
instrument manufacture Aerodyne®. The second dealt with best practices and feedback from the community. This second workshop also discussed the current evolution of the data uploading tool and the advances in the source apportionment analysis methods.

Figure 2.0 Comparison of time series, for ammonia (NH₄), sulphate (SO₄), nitrate (NO₃), and organic (Org). The black line in each time series represents the corresponding values given by the SIRTA reference instrument. The data in circles represent external tracers measured by other instruments.

2.2 Outcomes

The principle outcomes of this intercomparison were the development and improvement of inorganic calibration methods. The calibrations were initially recommended to be performed with a single inorganic species (Ammonium nitrate), now a mixture of two inorganic solutions are recommended; ammonium nitrate and ammonium sulphate (2:1 and 1:2) are recommended. Using this combination of inorganic solutions provides us with the advantage of calculating a robust instrument response to ammonium, as well as nitrate and sulphate species. The reproducibility of Q-ACSM instruments was addressed using the Z score analysis, following the recommendations by the international standard organization (ISO 5752-2). (Values within 2 and -2 are considered coherent and correspond to acceptable instrument performance. Values within 3 and -3 are considered questionable and need to be examined for additional problems. Values greater than 3 or less than -3 are not acceptable (Crenn et al., 2015, Thompson et al., 2006). This method provides a means to highlight any bias among the ACSM instruments. This provides standard procedures to follow for future intercomparisons (Fig. 2.1).
The standard operating procedures of these instruments were updated through discussion with the community (Appendix 1). The ACTRIS-2 ILC also provided the opportunity for different participants to discuss instrument issues and problems within the community to help share the knowledge.

As stated in the Grant Agreement, Task 3.2.1 will also conduct further ILC studies to support the continuing improvement of SOPs for calibration of organic aerosols.

3.0 Homogenisation of data processing and data submission

3.1 Data processing

One of greatest advantages within the ACSM community compared with other aerosol communities is the identical design of all the ACSM instruments. This has the advantage that the operating principles and mechanical design of each instrument is identical reducing the variability among measurements at different sites. This makes this sampling network unique within ACTRIS, since most other networks for aerosol measurements, gas-phase measurements, and vertical profiles have the added difficulty of comparing instruments with different operating principles and analysis software.

All measurement data obtained from the ACSM instrument are equally analysed using the same software, ensuring standardized and homogenized data processing. In addition to the standard analysis of aerosol chemistry data from the submicron data (Organic, sulphate, ammonium,
nitrate, and chloride), it is also possible to perform detailed source apportionment analysis on the organic mass spectra.

This analysis is currently performed with one of two statistical tools: Positive matrix factorization (PMF)(Ulbrich et al., 2009) or Source Finder tool (SOFI) developed by PSI using the multilinear engine-2 tool (Canonaco et al., 2013). There are yearly workshops organised by PSI around the SOFI tool to ensure that users are up-to-date on the latest changes and are aware of how the SOFI program functions. This ensures that users from different laboratories can maximise the amount of chemical processing obtained from their data.

3.2 Data submission protocols

The preparation of data uploading has been greatly simplified and homogenized through the development of the EBAS Export Tool for ACSM/AMS (EETA) uploader tool. This EETA tool has been developed by PSI (Jay Slowik) and facilitates the preparation of ACSM data for submission to the EBAS database. EETA allows the data format to be harmonized with respect to time and format. In addition, it is now possible to add in additional metadata to describe both the measurement site and the instrument type (Appendix 1).

There are also options to prepare and export organic mass spectral matrices for submission to the EBAS website for future analysis using SOFI or PMF. PSI is currently leading activities within ACTRIS to perform homogenous source apportionment of ambient organic aerosol obtained from several stations within the ACTRIS2 network.

This tool allows for simultaneous submission of L0 and L2 data to the EBAS server. The EBAS uploader tool is currently designed for the ACSM-Quad instrument, but will soon be updated to be compatible with the ACSM-ToF instrument. Currently data flags are limited to “9999: Missing measurement, unspecified reason”. These flags will be elaborated in the future to include addition information such as those used by the aerosol size distribution measures (see report WP3, D3.4).

4.0 Establishing and implementing on-going control procedures for ACSM instruments

After discussions during the ACTRIS2 ILC workshop, several means on-going control procedures were suggested. The simplest form of these control procedures was to gather calibration values for each instrument within the ACTRIS-2 network. As part of the SOPs, calibrations are recommended every 2 to 3 months. We made a request to users to record their calibrations (perhaps once every six months) and submit this data with the data submission to EBAS and additionally to the PSI/ACMCC. This will provide an in-expensive means of following instrument performance over time (Fig. 4). This data can eventually be made uploaded with the measurement data to the EBAS site.
Figure 4. Example of instrument calibration values taken over several months to observe changes in instrument response.

5.0 Future plans for the ACSM within ACTRIS2

The ACTRIS-2 WP3 Task 3.2.1 deliverables related to the ACSM measurements are planned to follow a logical chain of events that, at the end of ACTRIS-2, will lead us further towards our final aim of obtaining informative source apportionment of the organic aerosol at ACTRIS sites across Europe. It will also provide homogeneous and quality-assured data of online chemistry that can be used for development and evaluation of chemical transport models. The combination of a validated OA source apportionment and inorganic aerosol measurements can be used to support political decision-making that aims to safeguard human health and mitigate climate change in the EU member states.

The chain of events is:

i. Reporting on ILC1 for all ACSM instruments within the ACTRIS network.

ii. Assuring up to date SOPs for the suitable ACSM-Quad and ACSM-TOF instruments for subsequent implementation in ACTRIS.

iii. Establish procedures for the near real time submission of ACSM data to NILU.

iv. Determining dates and objectives for ILC2.

6.0 References


Appendix 1. ACSM Best Practices

This document contains recommendations for setup, operation, maintenance and data analysis procedures for the Aerodyne aerosol chemical speciation monitor (ACSM). It is intended as a supplement to the published ACSM DAQ and Igor manuals. Information in this reference is drawn from the ACSM DAQ and Igor manuals, the Aerodyne ACSM website (https://sites.google.com/site/ariacsm/home), the ToF-AMS wiki page (http://cires.colorado.edu/jimenez-group/wiki/index.php?title=ToF-AMS_Main), ACSM user experiences, and discussion during the May 2012 ACSM meeting at PSI.

Last update: 24.03.2015

1. ACSM Resources

ACSM DAQ and Igor manual Aerodyne’s ACSM website
https://sites.google.com/site/ariacsm/home
ToF-AMS wiki page
http://cires.colorado.edu/jimenez-group/wiki/index.php?title=ToF-AMS_Main

Site intended for ToF-AMS, but many issues are similar, e.g. collection efficiency, sampling line considerations, and instrument inter-comparison issues, and some operating/data analysis principles.

ACTRIS campaign website
http://www.psi.ch/acsm-stations/

2. Inlet and Sampling Lines

Construction of sampling system
The complete sampling system should be diagrammed, including sampling line lengths, diameters, materials, and flow rates.

Sample flow controller accessory (Aerodyne)
Several of the issues below (cyclone, pump/flow rate measurement) are facilitated by the sample flow controller accessory available from Aerodyne.

PM2.5 Cyclone on sampling inlet

Reduces clogging of the ACSM critical orifice and prevents transmission of large, high-mass particles. PM1 cyclones should be avoided because the PM1 size cut is then applied twice (at cyclone and at ACSM inlet). Further, a PM1 cyclone may introduce an RH-dependent transmission function. Deliverable WP21 / D21.6 (48) Version: 2015/03/268

Construct sampling lines from stainless and/or copper tubing
Conductive tubing materials reduce particle losses. Critically, **black conductive tubing (e.g. from TSI) should not be used**, because this can cause siloxane contamination of the mass spectrum, including at m/z 73 (an important marker for biomass burning). Stainless steel is ideal for long-term sampling. If copper is used, lines should be periodically checked for corrosion.

**Line losses and leaks**

Residence time in the sampling lines should be limited to reduce diffusive particle losses. This can be accomplished with (1) a pump (e.g. split from the Nafion dryer pump below) and/or (2) connecting other instrumentation (e.g. SMPS) near the ACSM inlet. Required flowrate depends on sampling lines, generally 1 to 10 L/min up to the dryer inlet. This flowrate should be continuously monitored and recorded.

The combination of tube width and sampling flow should be selected to maintain laminar flow (Re < 1000) to avoid particle losses from turbulent flow.

Important to test sampling line for leaks (e.g. filter on or near sampling inlet).

**Nafion dryer**

ACSM bounce collection efficiency is affected by relative humidity in ways that are difficult to predict. Note that it is the RH at the ACSM inlet that matters, not the ambient RH, and thus this effect is also influenced by outdoor/indoor temperature gradients. It is **strongly recommended to dry the sampled aerosol** (25 to 50% RH) before the ACSM inlet. This allows the collection efficiency to be estimated from the particle composition, and also facilitates comparisons with co-located instrumentation (e.g. SMPS).

Nafion dryers are preferred over silica gel dryers because they require less maintenance (silica gel dryers may require daily gel changes). The dryer should have a metal casing to reduce charged particle losses. The PermaPure MD series dryers work well:


The aerosol should be plumbed through the inner path, and a pump with critical orifice connected to the outer path to reduce the outer path pressure to ~0.1 atm. When connecting the inner path, care should be taken with the fittings to avoid breaking the internal seals on the Nafion membrane, introducing a leak to atmosphere. This can be checked by placing a filter at the dryer inlet and measuring zero particles at the dryer outlet with a CPC.

More information on dryer setup and operation is available at:
http://cires.colorado.edu/jimenez-group/wiki/index.php/Field_ToF-AMS_Operation#Dryer_Set_Up
2. Spare parts and support equipment

On-line uninterruptible power supply (UPS, 2000 kV)
Servo motor for inlet assembly

Filter for valve body assembly. Filter should be changed when a pressure drop is observed across the filter (see diagnostics plot in ACSM analysis softwa).

SEM (monitor the required voltage as a function of time to anticipate failure).

Filaments (2 sets of 2). At any time, 2 filaments are installed in the instrument. Should compare their performance after installation.

Critical orifice
Turbo pump
Diaphragms for backing pump and tool for changing diaphragms

Part numbers can be found at:
https://sites.google.com/site/ariacsm/support-information

3. Maintenance/Calibration Schedule

Continuously monitor instrument flowrate, signals for m/z 28, and turbo pump speeds/currents/temperatures. Stability of these parameters generally indicates stable ACSM operation. However, it is still advisable to regularly perform calibration/maintenance activities, particularly for a new instrument, at the beginning of a new deployment, or following venting of the instrument. For an instrument for which long-term stability has been confirmed, the calibration schedule can be reduced.

Daily
Monitor inlet pressure (~flowrate), m/z 28 and naphthalene signals. Significant changes indicate maintenance is needed.
Monitors turbo pump speeds, currents, and temperatures. This helps to anticipate pump failure.

2x per week
Check and record detector amplification (use N2 signal as reference: 1e-7 amps), adjust if needed (if change >15%).
Detector amplification should be checked on a daily basis if possible at the start of a campaign or after venting the instrument, until stable operation is observed. For new instruments the detector voltages have to be adjusted more frequently.

Every month (or as needed; this is highly site-dependent)
Clean dust from turbo pump fan filters & fan in the Prisma head.
Check flow rate, clean orifice with isopropanol, check flow rate again.

Every 2 months
IE calibration with ammonium nitrate & RIE calibration with ammonium sulphate. This should be done more frequently (weekly) at the start of a campaign or after the instrument
has been vented, until stable results (i.e. airbeam to IE ratio) are obtained. Deliverable WP21 / D21.6 (48) Version: 2015/03/26

**Every 3 months**
Collect data with (1) valve closed to check background (2) valve open with filter in-line. Several data points are needed, suggest collecting for at least 1-2 hrs at 15-min time resolution. Check inlet pressure with valve open and closed to evaluate vacuum/pumping integrity. Keep DAQ software updated (~6 months or following crucial update). DAQ is available from Aerodyne’s ACSM users’ site.

**Other**
Evaluate stability of m/z calibration and resolution. Normally this is stable, but should be individually confirmed.

### 4. Calibrations

#### Flow calibration
Critical to record the ambient pressure and temperature at which flow calibration is performed.
Necessary to adjust critical orifice size if instrument is operated at altitude.

#### Ionization efficiency
All IE calibrations should be immediately preceded by tuning of detector voltage and heater bias voltage.
It is important that the NH4NO3 calibration solution not be more concentrated than recommended (5 mM) to avoid artifacts from multiply-charged particles.
Confirm that particles are **dried** before size-selection.
Measure ions for NO3+ (m/z 30, 46), NH4+ (15, 16, 17), and air. These are the standard settings if the custom scan file for calibration with NO3NH4 from the Aerodyne ACSM webpage are used. For calibration with ammonium sulphate use the respective custom scan file also available on the Aerodyne webpage.
Ratio of m/z 28 to IE should be **logged and monitored**. This serves as a reference point for sensitivity corrections during data analysis.

#### Relative ionization efficiency
SO42-: Should be measured immediately following an IE calibration, where the RIE of NH4+ has just been calculated from mass balance between NH4+ and NO3-. Nebulize (NH4)2SO4 and sample with the ACSM. The RIE of SO42- can then be calculated from mass balance with NH4+, since NH4+’s RIE is known. This method is not biased by particle bounce.
Make sure, that the ammonium nitrate calibration is performed before the ammonium sulphate calibration. For the calibration with ammonium sulphate start with small concentrations and increase Deliverable WP21 / D21.6 (48) Version: 2015/03/26 11
slowly. Due to partly slow evaporation of ammonium sulphate hysteresis effects can influence the calibration if done otherwise.

Methanesulfonic acid (MSA): It is advisable to calibrate for MSA if sampling near the ocean. This species has been observed in previous ACSM/AMS measurements but the RIE is not well known.

5. Operation and analysis

Sampling time

30 minute time resolution is generally recommended (e.g. 1 open 1 closed 28 sets at 200ms/amu). Maximum of 1 hr for sites with very low concentrations. 15 minutes can be very helpful if local sources are important. Note that short sampling times can easily be re-gridded during post-analysis to generate longer averages if signal-to-noise is problematic.

Collection efficiency (from particle bounce)

Uncertainties due to bounce collection efficiency ($E_b$) can be greatly reduced by installing a Nafion dryer as discussed above.

Bounce collection efficiency should be estimated using the composition-based estimate given in

Middlebrook et al. (2012). Further investigation by comparison with external instruments (e.g. SMPS) is strongly recommended. Note that the Middlebrook et al. method was developed using AMS data, but applies equally to the ACSM. It requires (1) an estimate of the ammonium detection limit, and (2) when ammonium is below detection limit, an estimate of minimum collection efficiency ($E_b = 0.45$ is recommended). Middlebrook, A.M., Bahreini, R., Jimenez, J.L., and Canagaratna, M.R.: Evaluation of composition-dependent collection efficiencies for the Aerodyne aerosol mass spectrometer using field data, *Aerosol Sci. Technol.*, 46, 258-271, doi:10.1080/02786826.2011.620041, 2012.

http://cires.colorado.edu/jimenez/Papers/2011_AST_Middlebrook_CE.pdf

When investigating collection efficiency by comparison with co-located instruments, care should be taken to assess assumptions/approximations used to perform such inter-comparisons! Note also that other instruments have their own uncertainties, and disagreements with the ACSM may not necessarily be due to collection efficiency. **Collection efficiency should not be a catch-all for disagreements between instruments!**

A few points relating to comparisons of ACSM with SMPS (or similar instruments):

(1) Particles should be dried for both instruments
(2) Transmission ranges of the ACSM and SMPS should be synchronized, especially at large particle sizes (can be done during SMPS analysis, as long as the large-size SMPS cutpoint exceeds that of the ACSM) Deliverable WP21 / D21.6 (48) Version: 2015/03/26

(3) Refractory species (black carbon, dust, sea salt, etc.) must be negligible or measured by other methods with comparable size transmission.

**Sensitivity/gain corrections during analysis**
Recommended to use m/z 28 signal (rather than naphthalene) for gain/sensitivity corrections due to better signal-to-noise. Recommended m/z 28 reference value = 1 x 10^-7 amps.

**Post-Analysis (PMF, ME2, etc.)**
Filter outliers/bad points out of data before analysis (e.g. using continuously monitored data in section 3).

**Fragmentation diagnostics**
Org44 vs. Org43: Org44 has potential air interferences, while Org43 is an approximate (interference-free) surrogate for total organics. The Org44/Org43 plot should have an intercept at the origin. Otherwise fragmentation tables may need adjustment.

**Nitrate and sulfate diagnostics:** Check for (1) linearity; (2) intercept at origin. If these criteria are not met, fragmentation tables may require adjustment. Older versions of the DAQ software may have some nonlinearity (especially for nitrate) due to detector settings; fixed in newer software.

**PMF Matrix**
Use software version 1.5.3.5 or newer to create PMF matrices (older versions have bugs in the calculation of the error matrix).

PMF matrices should not include m/z > 120 because of large ion transmission corrections and naphthalene interferences.

The dwell time requested by export routine is the total integration time for a single m/z within a single saving interval (i.e. can include multiple scans of the mass spectrum). It is calculated as:

\[ \text{Dwell time} = (\text{number of MS scan sets}) \times (\text{integration time for 1 m/z in 1 scan}) \times (\text{stick integration width}) \]

Where:

- 1 MS scan set = open MS + closed MS
- Integration time for 1 m/z in 1 scan is determined from scan speed setting in DAQ
- Stick integration width = 0.1 amu (written in procedure as 2*ka_amu_window)

Recommendations for ACSM PMF analysis can be found in:


Appendix 2

A2.1 EBAS uploader tool.