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SCIENTIFIC REPORT

ESF Exploratory Workshop on

Mitigation of methane emissions through microbial oxidation on landfills - evaluation and quantification approaches

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Executive summary:

Landfills represent an important source of the greenhouse gas methane. During the past years it was shown in many international studies that enhancing microbial methane oxidation in suitable landfill covers (called biocovers) or biofilters is a simple and low-cost measure to mitigate methane emissions. The oxidation of landfill methane is a microbially mediated process, where specific bacteria, called methanotrophs, convert methane into water, carbon dioxide and biomass. This process is impacted by a lot of environmental conditions, like soil properties, water content, methane load, seasonal temperature changes, etc., which consequently affect the oxidation capacity and the actual oxidation efficiency. Due to some new European guidelines, increasing demands of governmental authorities, and to contribute to national and international greenhouse gas inventories as well as to asses the role of this mitigation strategy in carbon trading schemes it is important to prove the efficiency of biocovers and biofilters and similar systems to quantify methane oxidation activity and/or the remaining emissions.

The application of such bio-based oxidation systems requires the use of measurement methods and evaluation approaches, needed both in the planning stage as well as throughout the operation in order to prove their efficiency. Different strategies exist for the monitoring of the in-situ oxidation performance and efficiency in the field: either the remaining emissions and thereby the mitigation effect can be measured when compared to reference values (= indirect approach), or the oxidation activity of the microbes and its rate can be assessed directly in the soil or substrate layers.

Principally, for the first approach different techniques commonly used in monitoring landfill surface emissions can be applied to control methane oxidising systems. The evaluation of the effectiveness of actively vented and closed biofilter systems is comparatively easy. Inflow and outflow methane concentrations as well as flow volumes can be monitored (often online) quite easily and thus are well known. The quality control of open, passively vented systems can be checked easily as well, e.g., using a FID (Flame-Ionisation-Detector) unit to detect surface methane concentrations that may indicate leaks, fissures or insufficient oxidation. In contrast, measuring and quantifying emission fluxes in open oxidation systems is considerably more intricate. The determination of the overall effectiveness of such systems is, however, very complex, since the methane influx (reference flux or emission) into the system must be known. In some instances, landfill gas production data for a specific site can be surveyed (e.g, reference measurements of baseline emissions prior to the application of the system or simultaneously on adjacent cells), calculated or modeled. However, in most cases existing models reflect the actual landfill gas generation and emission situation extreme inadequately. Moreover, due to temporal and/or spatial variability, noticeable discrepancy can occur between reference influxes and day-to-day emission values. Thus, such indirect evaluation approaches are typically very laborious, comprise great uncertainties and need complex and comprehensive interpretations.

On the other hand, there are a lot of new approaches under research focusing on the direct evaluation of the methanotrophic activity and microbial oxidation efficiency. The composition of methanotrophic population can be identified and its activity can be estimated by several methods including e.g., biochemical tests, methane oxidation assays, enumeration methods, and molecular biology techniques. However, these methods are

often insufficient for monitoring microbial activity in complex, mixed microbial communities under alternating environmental conditions such as those present in landfills and landfill covers.

A promising approach and currently one of the most precise methods available for directly determining methane oxidation in landfill cover soils is represented by the measurement of stable isotopes. However, to date the method has displayed several limitations caused by variations in methane stable isotope ratios as a consequent to an extremely specific fractionation due to individual characteristics of methanotrophs and growth condition, fractionation processes during gas transport, as well as different isotopic signatures of the produced methane controlled by anaerobic formation processes.

The impulse-presentations during this exploratory workshop were focusing on both indirect and direct approaches. The first part of Thursday 13th December was filled with presentations on microbiological and lab-scale methods, including microbial diagnostic micro-arrays, phospholipids fatty acid analysis, FISH, in-situ microelectrodes, and lab scale capacity tests. The second part of the workshop day was dealing with field scale approaches to measure remaining methane emissions and to evaluate methane oxidation performance, including gas generation modelling and gas flux baseline monitoring, micro-meteorological methods and chamber/tunnel technologies to measure emission fluxes, in-situ oxidation activity tests and stable isotope techniques. On Friday, 14th December, small groups were formed to discuss and draft conclusion concepts regarding the suitability of all presented approaches and technologies for evaluating methane oxidation performance. After a general discussion of the drafted concepts, ideas were gathered for further cooperation activities, maybe within the scope of networking programs under the auspices of ESF.

Scientific content of the event:

The workshop focused on approaches to quantify and evaluate methane oxidation performance and efficiency in different oxidation systems on landfills, like conventional landfill cover soils, engineered biocovers, bio-windows and biofilters. Determining the oxidation activity and efficiency on landfills is crucial also for evaluating the emission situation and controlling greenhouse gas (GHG) release of a landfill site. Respectively, this process is an important measure in global warming mitigation strategies, and thus must be quantified.

The workshop started with a welcome session on Wednesday, 12th December, including the presentations of Sonja LOJEN, ESF-representative, and Jean BOGNER, Coordinating Lead Author of the IPCC AR4 Working Group III – chapter 10, who gave an overview from the IPCC 4th Assessment Report with respect to the significance of waste management measures, landfill gas emissions and methane oxidation among other worldwide greenhouse gas emissions and mitigation strategies, respectively. She pointed out that for the first time a separate chapter on waste has been included in the 4th IPCC assessment report, showing the increasing importance and high potential of waste management measures to contribute to global warming and its mitigation strategies to counteract this issue. Total GHG emissions from the waste sector are only 3% of all global GHG emissions, but almost 20% are contributing to global anthropogenic methane emissions, dominated by the microbially produced methane in landfills as the largest source. Landfill methane emissions are stabilizing or decreasing in many developed countries due to landfill gas recovery or decreased landfilling of organic waste (particularly true for many European countries), but landfill methane emissions are increasing in developing countries. The IPCC numbers show that landfill methane emissions will increase from about 520 – 750 Mt CO_{2equ} calculated for 2005 up to 820 – 1000 Mt CO_{2equ} in the year 2020. Actual IPCC -strategies for mitigation of GHG emissions from the waste sector includes: landfill CH4 recovery and utilization, optimizing methanotrophic CH4 oxidation in landfill cover soils and biofilters, avoidance of GHG generation on landfills via source separating, composting, incineration (and other thermal processes) or mechanical and biological treatment, and avoidance of waste generation (e.g., recycling, re-use, waste minimization).

The second workshop day was filled with key-note presentations dealing with different approaches to evaluate methane oxidation performance from lab scale to field scale. The day was opened with a short introduction and **overview on** different technologies and strategies by Marion HUBER-HUMER. Different **strategies** exist **for the monitoring of the oxidation performance and efficiency in the field**: either the remaining emissions and thereby the mitigation effect can be measured when compared to reference values (= indirect approach), or the oxidation activity of the microbes and its rate can be assessed directly in the soil or cover layers. The functionality and significance of applied technologies and the suitability of diverse indirect or direct approaches are depending on

- landfill site characteristics (size, waste input data, geometry,...)
- the oxidation system that shall be surveyed and evaluated (closed / open, small/large extension,...)
- the properties of the material used for methane oxidation (compost, fine/coarse soils, artificial compounds,...)
- the aim and scale of the survey study (field or lab scale, determination of emission mitigation effect or microbial oxidation activity,…)

Currently, every approach available for evaluating methane oxidation hold strengths and drawbacks, thus technologies and evaluation strategies have to be adapted and modified for the specific application situation of the survey to minimize the disadvantages. A main general problem of indirect approaches (evaluation of mitigation effect via gas emission measurements) is to determine reference and baseline values, which is typically very laborious, comprises great uncertainties, requires multi-disciplinary approaches as well as complex and comprehensive interpretations.

During sum-up and discussion following main research question arose**:**

- *How to determine reference values for the evaluation of oxidation efficiency or methane emission mitigation effect, respectively?*

After the introductory presentation, five key-note presentations followed in the morning session of the second workshop day, dealing with different technologies to evaluate and quantify methanotrophic activity. Nancy STRALIS-PAVESE talked about the principals of **diagnostic DNA- and mRNA based micro-arrays** for analysing methanotrophic bacteria. She pointed out that this method offers a fast, parallel detection and identification of methanotrophs with high resolution, however, one big drawback is that probe stets are only limited to microorganisms with already sequenced genes. She concluded that the detection and the relative signal for a detected bacterial group is due to the abundance and the metabolically active members of that group (mRNA level). Moreover, her investigations show that quantitative PCR and quantitative RT-PCR results are in agreement with the microarray results.

The follow-up discussion was focusing on the practical applicability of micro-arrays for landfill covers. Participants tried to summarize research needs and the current opportunities offered by this method for the specific application on landfills :

- *We can determine "which strains grow, which do not"*
- *We can assess which strains are important for the investigated community and what is "going on " in the system*
- *Micro-arrays can be a powerful tool to evaluate a biological system (qualitatively!) – but the time factor must be considered: the data are only snapshots of current oxidation activity (in soil samples RNA degrades within minutes; DNA can persist for weeks and longer)*
- *Suitable method for comparison of different substrates/materials and for pre-selection of materials for landfill cover/biofilter construction*
- *Micro-arrays should be coupled with other methods; e.g. to correlate results from oxidation capacity tests with micro-array analyses – not enough data for correlations at the moment!*
- *Micro-arrays should be investigated under changing conditions (including different methane fluxes, concentrations,…)*
- *Micro-arrays seem to be a good supporting tool, but not a single method to evaluate methane oxidation efficiency in landfill covers at the moment – further development and validation is clearly needed!*

Gunnar BÖRJESSON continued with a presentation on a Swedish project where **different methods and approaches had been combined to quantify the action of methanotrophs** in landfills to gain data for landfill emissions, methane oxidation in the field and gas production. Traditional gas emission measurement methods were coupled with innovative methods, including also isotope and microbiological approaches (PLFA). The overall aim was to quantify the role of biological oxidation in the global methane budget. The problem with stable isotope techniques is the determination of the fractionation factor (which in their study turned out to have a strong dependency on temperature), and that this method can hardly be applied when methane is oxidized completely in the cover. A further conclusion deduced from their investigations was that PLFA data could not be correlated with emission measurement results.

The subsequent presentation by Andrea WATZINGER focused on the quantification of methanotrophs by **phospholipid fatty acid analysis (PLFA)** including a 13C isotopic approach. She explained in detail the PLFA method including 13C labelling at natural abundance, e.g. using the natural difference of the ratio of 13C/12C of the biocover and methane from the landfill. The arising small differences between e.g., compost (δ13C = -30 ‰) and methane (δ13C = -50 ‰), and the fractionation in the microorganisms may be practical limits on detecting and quantifying the fate. There appeared also instrumental problems, like the loss of resolution during GC-c-IRMS measurements. Moreover, knowledge about the life time of microbial biomarkers from dead cells (DNA, RNA, PLFAs, quinones, sterols...) seems to be crucial. There seems to be better applicability for Type I than for Type II methanotrophs (mismatch of abundance and labelling – maybe due to the use of other substrates?). However, by isotopic labelling of PLFAs the presence of an active methanotrophic community in a cover can be confirmed, secondary turnover of carbon can be followed (foodweb), and unknown microorganisms and mechanisms can be identified.

The follow-up summary and discussion addressed mainly the opportunities of this method for field application:

- *For field application a stronger labelling would be needed (financial and experimental restrictions!); currently, the method is more suitable for lab scale studies)*
- *Is the combination of PLFA and the stable isotope method conceivable for quantification the field? – At current knowledge, one main problem will be the discrimination factor, which varies with field conditions.*
- *Currently, PLFA provides the possibility to quantify the methanotrophic biomass but not its effect on the methane oxidation rate!*

General principals of other **micro-methods** including very new approaches were presented by Piet LENS, like FISH, in vivo 13C nuclear magnetic resonance spectroscopy, DGGE, Raman spectroscopy and specific micro-sensors. Most of these methods provide an opportunity to identify methanotrophic microbes and can go deeply into detail, but gives sparse functional information. Therefore, the combination with batch tests or other activity assessment methods is needed. Micro-sensors, like microelectrodes to measure consumed oxygen, can provide micro profiles within a medium and may give information on the methanotrophic activity (oxygen consumption); but currently such micro-sensors work only

in water saturated medium, thus not (yet) well adapted for the application in landfill cover environments.

In the sum-up participants concluded that,

- *currently, all these methods provide a high potential for determining methanotrophic communities on lab scale, but there is a big gap for application in the field and further research and development is needed;*
- *most promising methods for the application on complex environmental samples like landfill covers are the Raman Spectroscopy and micro-sensors (at the moment only available for measurements in the water phase)*

The last presentation in the morning session was given by Julia GEBERT, who talked about the standardisation of **methane oxidation capacity tests**. Batch tests are a widely accepted, cheap and easy to conduct method to assess and compare the methane oxidation capacity of different substrates. However, there is no existing standardised procedure, thus the comparison of batch data from different laboratories is hardly possible and reliable. The need to harmonize this method and to develop a robust, easy-to-use protocol is evident. A general problem with batch test is the destructive sampling procedure (leading to a strong change of all physical soil properties) and the small input amount. Possible influencing parameters are: sample treatment, water content, initial gas concentrations (CH4 and O2), incubation temperature, flask size, amount of tested material, and data interpretation. As an example, a different sample preparation with respect to water content (in-situ water content versus slurry) both tested in a sandy and loamy material led to totally different results depending on the material properties (higher oxidation capacity in sandy substrates at in-situ water content, higher capacity of loamy substrates in a slurry). An obvious problem is the gap between batch data (oxidation capacity of substrates) and the ”reality”, meaning the real actual oxidation rate in the field or even measured in laboratory soil columns. Batch assays can be used to compare a potential, but not to estimate field performance of a specific substrate. An assessment of a trend within a cover profile as well as the estimation of methanotrophic abundance in an actively oxidising soil sample is possible.

Following questions and conclusions emerged during discussion:

- *Is it possible to give a reliable relation of batch results and activity in the field?=> At the moment only the comparison of active soil samples from the same location or from soil profiles is reliable (only snapshots!). It is not reliable for material selection or to get information an practical application. Column tests are more suitable to answer the question what to use and how to design your cover material.*
- *It would be favourable to work with undisturbed soil cores (excavated from existing covers) in batch assays.*
- *There exists great differences in the suitability and applicability of batch assays depending on the material to be tested (e.g, soil or compost, grain size, gas permeability of substrates, etc.)*

- *We have to keep in mind that for scientific purposes and process understanding batch tests may offer a good tool, from a practical point of view and for field evaluation this method holds strong limitations.*

In the afternoon session the focus was on field scale approaches to evaluate methane oxidation efficiency. The first key-note speaker, Heijo SCHARFF, presented different **methods currently applied to estimate and model methane production, oxidation and emissions** on landfills, particularly from a practical point of view. Countries that have ratified the Kyoto-protocol make National Inventory Reports for UNFCCC, in which annual emission of all GHG gasses is reported. Thus, both operators and national authorities need methods for estimation of annual landfill methane emissions. By means of a landfill gas mass balance he pointed out the defaults and weakness of the current status of estimating, measuring or modelling the different parts (methane oxidation, recovery, production) in methane emission calculation as well as the advantages and disadvantages of different emission measurement methods, like chamber measurements, plume methods (static and dynamic), and micrometeorological measurements. Methods with high spatial and temporal resolution are needed. Plume and micro-meteorological approaches seem to be most suitable to assess whole landfill emissions. The oxidation factor currently used in most mass balances is a generic value (0 for uncovered landfills and 0.1 for covered landfills), but does not reflect reality (oxidation factor of 0.2 - 0.4 is not uncommon). He concluded that landfill methane emission estimation methods currently do not meet E-PRTR requirements concerning consistency, authenticity, reliability, comparability and transparency. Particularly models must be harmonised and improved, but it is very unlikely that existing models can be made significantly more accurate for landfill estimates. Emission measurement methods need further development, need to be more practicable and affordable and validated. Practicable methods to estimate methane oxidation on a landfill site are required. From his point of view, it can not be considered useful that new methods for emission and oxidation estimation will have a better than ±20% accuracy.

In the sum-up participants summarized following questions and conclusions:

- *The different models to calculate methane production and emissions must be harmonized – field measurements could be used to improve model parameters. Currently, there exist big differences between model results.*
- *The accuracy of landfill mass balances is estimated to be +/- 500 % at current status and set of methods.*
- *Chamber methods are not suitable for whole landfill emission evaluation; they usually underestimate real emissions.*
- *The applicability of plume and micro-meteorological methods depends on site conditions (landfill geometry, vegetation, surrounding landscape,…). Micrometeorological methods often underestimate emissions compared to plume methods (about 30% difference).*
- *The combination of different methods would be favourable to evaluate emission fluxes and pattern on landfills.*
- *There is need to develop and combine emission measurement methods with oxidation efficiency evaluation.*

Jean BOGNER continued with a presentation on **new approaches for field quantification of methane oxidation**. She also addressed scaling up and new modelling approaches and presented strategies for improved regional GHG inventories including methane oxidation by means of an US example, an ongoing project in California. Goal of this project is to develop a field-validated annual model for landfill methane emissions inclusive of oxidation. Currently, in the US the stable isotope method in combination with methane mass balance is the preferred approach to estimate methane oxidation in the field. The method can be applied at different levels: above ground (upwind and downwind), at ground level (comparison between emitted methane and anaerobic methane inside the landfill), and below ground (from soil gas profiles). In former days (prior to stable isotope technique), simple field incubations in parallel with emission techniques have been applied to assess methane oxidation, gaining stable results but very time-consuming. Model development must move toward more inclusive, field-validated, ecological process modeling for landfill methane oxidation and emissions. There is need and potential for "Cross-fertilization" between models via development of modular sub-model approaches (e.g., for production, transport, oxidation, and emissions).

Questions and sum-up were focusing on

- *Which parameters should be included in such models? Vegetation – how can it be considered? Influence of pressure and temperature should be combined.*
- *Which data are required to calibrate and validate the models?*
- *In the US project emission fluxes are measured on different cover materials and types to calibrate models – daily operating areas (young waste up to 30 days old) are already producing a lot of methane.*

Peter KJELDSEN presented a concept to evaluate methane oxidation efficiency in biocover and bio-windows systems focusing on the **determination of methane loads under field conditions**. He talked about the approaches applied and developed in an ongoing Danish project ("BIOCOVER") for evaluating local methane fluxes and loads going into the oxidation systems. The information on methane loads is crucial for the evaluation of all technologies. Methane load for full sized mitigation systems may be affected by large scale heterogeneities (hot spots, emission from leachate collection system). In the Danish study a baseline survey (10 measuring campaigns during a 1-year test period) was conducted prior to the installation of the biobased mitigation systems and followed by methane measurements subsequently to the construction of the biowindows. Thus, the overall mitigation (= oxidation) efficiency is calculated valid for the measured methane load on large scale. Methods to test the efficiency and methane load on local scale includes for example deep flux chambers (vented to avoid bias from pressure build up), pore gas velocity measurement by tracer release, carbon balance or methane balance (based on stable isotope signature). The carbon balance method includes detection of surface carbon fluxes (q_{CO2}+q_{CH4}), estimation of surface carbon flux contribution from compost/soil respiration (q_r mainly based on lab data), measurement of CO2 and CH4 concentrations in deep gas (C_{CH4} and C_{CO2} in vol%), and assumes stationary conditions.

Methane load (mol/m^2·day) is then calculated according to

$$q_{CH_4,load} = \frac{C_{CH_4}}{C_{CH_4} + C_{CO_2}} \cdot \left(q_{s,CO_2} + q_{s,CH_4} - q_{r,CO_2} \right)$$

Methane load on a local scale is an important reference to gain basic process understanding under field conditions. There is a big need to develop methods for evaluating local methane loads.

Participants summarized following conclusions and research questions:

- *What is an appropriate soil gas velocity tracer? – CO or fluorocarbon?*
- *Many measurements are needed for reliable background data and the baseline stud. => time consuming investigation.*
- *The measurement of CO2 fluxes together with methane emissions is favourable to get additional information, more reliable data that can be better interpreted.*
- *The determination of soil respiration activity, particularly in organic rich substrates like composts is required. E.g., in lab respiration tests or in a box (without landfill gas input) beside the oxidation system under the same environmental conditions in the field, where CO2 emissions (basic respiration) can be measured and calculated.*

Tuomas LAURILA introduced and explained the **micrometeorological Eddy-covariance method, a remote sensing method**, to measure methane and carbon dioxide emissions. Compared to the tracer method, it provides spatial information, CO2 and H2O fluxes are measured together with CH4 fluxes, and gas recovery efficiency and methane oxidation rates can be calculated from the field data. Surface oxidation may be calculated as
$OX = 1 - E_{CH4} / (\text{fraction}_{CH4} \times E_{CH4+CO2})$, where fraction_{CH4} is the fraction of CH4 in landfill gas, E_{CH4} is the emission of CH4, and $E_{CH4+CO2}$ is the emission of CH4 +CO2 under the assumption that no aerobic carbon decomposition occurs in the surface layer, and the methanotrophic microbes are not a net sink of carbon. However, the estimation of aerobic decomposition in the surface cover is possible when calculated as

$$OX = 1 - E_{CH4} / (\text{fraction}_{CH4} \times (E_{CH4+CO2} - E_{surfCO2})),$$

where fraction_{CH4} is the fraction of CH4 in landfill gas, E_{CH4} is the emission of CH4, $E_{CH4+CO2}$ is the emissions of CH4 +CO2, and $E_{surfCO2}$ is the emission due to CO2 decomposition in the surface layer. In their study, CO2 respiration was measured in static chamber using LiCor7500 open path CO2/H2O analyzer in 4-25 deg C. Within this investigation different oxidation factors were found depending on seasonal conditions, landfill geometry and cover type ranging from 0.1 (late autumn) to 0.4-0.5 in late summer on a compost-soil surface sloping area, and an oxidation factor of 0.1-0.17 during spring on a landfill top area with daily cover only.

Following questions and conclusions emerged:

- *If we monitor emissions for climate convention purposes, is it strictly the methanotrophic bacteria oxidation which we are looking for or is it the net effect to the atmosphere which is relevant?*
- *Comparison with other methods, like the stable isotope approach, is desired and will be further research work.*
- *What is the effect of vegetation?*
- *Variations of the background concentration do not contribute – difference between upwind and downwind is measured (covariance).*

Marion HUBER-HUMER gave an overview from an Austrian biocover project where a **3-step concept for the evaluation of methane oxidation efficiency** of differently designed compost covers was performed including (1) lab investigations for material selection (counter gradient column tests for methane oxidation potential, substrate characterization and molecular investigations (PCR, DGGE)), (2) qualitative evaluation in the field (methane surface concentration pattern with FID) inclusive determination of environmental factors and soil properties, and (3) quantification of mitigation effect in the field (using an open flux tunnel to measure CH4 and CO2 fluxes). To compare the methane mitigation efficiency of different biocover designs, flux emission data from the biocovers were compared to emissions released from adjacent uncovered reference cells under the same test conditions. A mean “area weighted emission rate” was calculated for each biocover and the uncovered landfill cell, by grouping the concentration data from the FID-measurements (categories). A mean annual flux rate for each category was then calculated from several (at least three to five reliable measurements for each location) tunnel placements over a one year investigation period. Based on the FID-mappings, the surface dimensions (m²) of the different categories were determined (= emission area) and combined with the corresponding methane fluxes. The findings from a two year investigation indicate a good correlation between the FID data and the flux measurements taking similar boundary conditions for measurements into account. This evaluation approach was for scientific purpose gaining a lot of information and data, is feasible with common and easily available, low-cost equipment, but was labour intensive and required a complex and comprehensive interpretation. Suggestion for practical application: Intensive investigation program over about one year (= „initial check of proper operation“) of the cover system, e.g., including FID measurements (1-2/month), flux measurements (1-2/month), gas/temperature profiles (2/month), determination of cover parameters (2/year). After statistical evaluation (correlation of data), a slim “routine monitoring program” can be followed up based on less labour–intensive methods.

Discussion and summary focused on

- *variability of concentration pattern and flux measurements => good correlation between FID concentration data and corresponding flux data (over one to two years investigation period); there was no great changes in FID spatial pattern (only slight temporal variations in concentrations)*

Julia GEBERT introduced a new approach to measure in-situ methanotrophic activity in the field, the **gas push-pull test** (GPPT). The GPPT for methane oxidation is an adaptation from the push-pull test used to quantify microbial activities in aquifers (e.g. denitrification, sulphate reduction). During a GPPT, a defined volume of a gas mixture containing a reactant (CH4) and a conservative tracer (e.g. Ar, Ne) is injected into the soil at the depth of interest using a pump and a perforated tube. After a desired incubation period, the mixture of reactant, tracer and soil gas is extracted from the same tube. During extraction, the gas mixture is sampled periodically in order to obtain breakthrough curves of reactant and tracer from which reaction rate constants and mass balances can be calculated. The extracted concentrations of reactant and tracer have to be corrected for background concentrations in the soil gas atmosphere. Consumption of reactive gases occurs during injection and extraction (transport dominated by advection) as well as during the incubation phase (transport dominated by diffusion). Important prerequisites for a GPPT are that the injected gas mixes well with the soil gas and that tracer and reactant transport behaviour is similar (e.g., Ar seems to be more suitable than He as it has similar diffusive transport behaviour as CH4). The latter is particularly relevant at longer incubation times during which transport is dominated by diffusion. She pointed out that GPPT bears great potential for the quantification of in situ methane oxidation in landfill cover soils. Critical points include the estimation of the spatial resolution which varies with soil texture and water content, the effect of diffusion and advection, the concentrations of CH4 and O2 to be injected, the injection/extraction rate, incubation phase duration, injection volume, and relating oxidation rate constants to a defined soil volume / mass. The procedure is currently being evaluated for application on landfills and tests are expected to be run on five old landfills in 2008 within the frame of the “MiMethox research project”.

Research questions and discussion emerged on:

- *What about measurements in shallow depths (30 - 40 cm) => interference with atmospheric air?*
- *Need for correction of background concentrations in the soil (methane and tracer) – problems if soil concentrations are initially high.*
- *How dealing with higher CH4 concentrations – is zero/first order kinetics appropriate? => The high dilution effect allows first order kinetics.*
- *The concentration gradient between the (noble) tracer and methane will vary if there is methane initially present in the soil but no tracer gas.*
- *Advection is induced when operating at high injection rates – influence must be considered?*
- *Online monitoring would be desirable - limiting factor is often the parallel detection of gases and tracer (e.g. Ar).*
- *The gas push-pull test is a promising tool to detect what is going on in the methane oxidising landfill cover.*

The last two presentations on the second workshop day focused on the stable isotope approach. Koenraad MAHIEU explained the principle of the stable isotope method and subsequent modelling approaches to quantify methane oxidation. Bacteria oxidize the lighter $12CH_4$ isotope slightly faster than the $13CH_4$ and $12CH_3D$ isotope leading to a fractionation factors (α_{oxC}) ranging typically between 1.01 – 1.03. The fractionation factor is calculated from batch experiments in the lab, isotope ratios of the produced (dA) and emitted (dE) CH_4 are measured in the waste and at the top of the landfill, and fraction oxidation is calculated according to

$$f_{ox} = \frac{\delta E - \delta A}{1000 \cdot \alpha_{ox} - 1}$$

The advantages are that it is a non invasive method allowing direct measurement of methane oxidation, and that sample collection is easy. A current big drawback is the high variability of the fractionation factor, so that it must be determined specifically for each investigation site (laborious incubation tests). Moreover, other factors are supposed to influence the isotope fractionation, like temperature and diffusion, maybe leading to an underestimation of microbial oxidation. Further critical points that may lead to underestimations are that the methane flow through macro pores (which is less oxidized) contributes more to the emitted methane sampled at the surface, and completely oxidized methane is not represented at the surface. A better approach could be to take gas samples for instance in 10 cm depths using probes. Hydrogen fractionation is 10 times higher than C fractionation which may give a better estimate on methane oxidation, since the fractionation is not as strongly influenced by diffusion. Moreover, there exist new modeling approaches including stable isotopes and diffusion fractionation to better estimate methane oxidation. Koen MAHIEU concluded that isotope fractionation is a promising technique but accuracy should be improved. Maybe, the addition of probe measurements and hydrogen fractionation can give a better estimation of CH4 oxidation. If general rules for the fractionation factor could be found, this method would become more practicable, since laborious incubations for each landfill site is no longer required.

Charlotte SCHEUTZ followed up with a presentation on a Danish field trial where the stable isotope method is currently applied to test the oxidation efficiency of biocovers/biowindows. In their study two approaches are applied: (1) measuring whole site oxidation: Stable carbon analysis are done on the gas generated within the waste, and on ambient gas sampled up-wind and down-wind of the landfill site; and (2) bio-window oxidation: Stable carbon analysis are done on the gas samples from flux chambers (surface and deep) and gas probes installed in the compost cover. She concluded that the isotopic approach gives the relative effectiveness of cover mitigation (the fraction of CH4 oxidized, %) but not the total mass of methane oxidized. In their study surface measurements and profiles do not give similar results, there occur high variance. So which approach should be used? The fractionation factor (α-value) is a critical parameter in determination of the oxidation fraction. If surface fluxes and gas concentrations are low, $d^{13}C$ can not be analyzed due to detection problems. The applicability of the isotope method is also critical when the oxidation system is working well, meaning high oxidation rates and very low or no methane fluxes at the surface or upper layers.

The subsequent discussion and conclusion focused on

- *The variation of the fractionation factor which varies strongly between landfill sites; more precise knowledge about the parameters influencing this factor is needed to get an idea how to determine it. There are indications that the fractionation factor is depending on the methane concentration – at low concentrations it is decreasing towards 1.0.*
- *Metabolic pathways and temperature are further influencing factors on the isotope fractionation.*
- *The fractionation factor might vary with scale and investigation design – in batch tests the factor will be different from column tests – and particularly in the field. But what we need is a value for the whole landfill site! Consider: in the field – we investigate an open system; in the lab a closed system!*
- *Background values must be taken into consideration when measuring a whole landfill site oxidation rate! In some studies different factors occurred at different distances from the landfill source, which seems to be a problem of the landfill set-up.*
- *The fractionation factor should be determined on a microbial species level, at least for groups Type I and Type II. Stable communities may be less susceptible to fractionation than specialized systems dominated by single species. However, the question remains open, if environmental conditions have a greater impact on the fractionation or population composition?*

After a short wrap-up on the third workshop day, four small groups were formed to intensively discuss the following issues:

First group:	Molecular and Micro-methods to quantify methane oxidation
Second group:	Stable isotope method – how to develop
Third group:	Emission measurement methodologies
Fourth group:	How to gain reference values for methane load

Each group was urged to reply to the specific questions that arose on the second workshop day during sum-up and discussions after each key-note presentation, and to draft a concept including a list of benefits and current drawbacks/limitations of each method/approach, statements the group agreed on, and recommendations for further development and future application. After the small group work the output from each group was presented to the whole auditorium and discussed jointly.

Conclusions from the group "Molecular and micro-methods to quantify methane oxidation":

method/approach	*benefits*	*questions*	*actions*
PLFA	- Can find new methanotrophs (does not rely on sequences) - Activity can be determined - Quantification possible together with isotope labelling → number of active methanotrophs	- Are natural isotope ratios suitable to be used for isotope labelling - Do different strains have different ;- values	- Measure turn-over-rate and isotope-discrimination
Hybridisation methods - FISH and microarrays	- Qualitative contribution to black box: FISH: spatial resolution, microarrays: high resolution below species level - Activity can be determined – RNA, or 14C, or 13C (Raman Microscopy) - Quantification possible in combination with quantitative PCR (number of RNAs per g soil)	- Can quantitative PCR-data be correlated with methane oxidation rate	- Optimise quantitative PCR and determine correlation with methane oxidation rates
Micro-sensors	- Measurement of direct activity with high spatial resolution - Can contribute to determine methane oxidation rate together with mass flow measurements	- Is high resolution needed? - How to integrate in a model on landfill scale	- adaptation to measure in unsaturated soil (miniaturisation of current gas probes?)

General Statement:

Benefits: All methods can contribute to a better understanding of the system and thus can in principle contribute to answer the question: "Why is it working, working badly, or working not at all"

Drawbacks/Limitations: Missing data to link them to determination of methane oxidation rate

Recommendation: Determination of turn-over rate of active methanotrophs is needed
Validation test among different methods (application of all methods on the same samples)

Conclusions from the group "Isotope method – how to develop":

Methodology - Modified calculation including fractionation by transport:

$$f_{oxo,z} = \frac{\delta_z - \delta_{anox}}{1000(\alpha_{ox} - \alpha_{trans})}$$

Where:
δ_{anox} and δz are standard 13C isotope ratios for the anoxic zone and sample at depth z or emitted CH4
α_{trans} is the isotope fraction factor due to transport (α_{trans} = 1 for purely advective transport, and α_{trans} >1 where diffusion is important)
α_{ox} is the isotope fraction factor due to oxidation

Benefits:

- The only method for direct quantification of methane oxidation in field settings (incl. both whole site and cover mitigation)
- Easy to sample (flux and plume samples are often sampled for emission measurements) and analyze (can do relatively low concentrations) if you have the equipment
- Easy (and cheap) method to apply if we can solve a number of problems…

Drawbacks:

factor - δ_{anox}:

- Needs to be measured for each site investigation
- Can be easily determined and variability is generally low within one site
- Might be variability in time (depending on the age/decomposition of the waste)
- Might be variability between sites (seems to be low)
- From a scientific point of view it could be interesting to investigate the dependence of waste type and degradability on the delta value

Factor - δ_z:

- Mainly question is how to sample gas for analysis
- Face similar problems concerning spatial variability as with methane emission measurements – but worse
- Need both approaches chamber/profiles and plume measurements depending on the purpose of your investigation
- More problems related to plume measurements…

Factor - α_{ox}:

- Very sensitive parameter – need to be determined as accurate as possible
- We don't understand the basic of processes influencing the fractionation during oxidation
- Currently best practice is to determine α_{ox} in batch incubation experiment under field temperatures. Lot of parameters will influence the measured fraction factor (initial CH_4 conc., growth phase or not, microbial community structure, temp., soil moisture content, sampling depth etc.). Currently we don't know the influence of many of these parameters => need for basic research
- We can measure the fractionation factor very well from an analytical point of view but we don't know what it represents

Factor - α_{trans}:

- Related to α_{ox} as it is a way to account for the fractionation due to diffusion
- The α_{ox} – value obtained in batch experiments include the effect

General statement:

- An overall question of cause is - whether the equation is representative of what is really going on in methane oxidizing covers
- Still expensive to analyze samples – method is not directly available for landfill operators as an practical/routine application (not yet) – more or less still a tool for research studies
- Need some level of special knowledge about isotopes to interpret the data – the process is not completely understood yet, there are many unknown factors influencing the system to large extent
- It only gives a percentage of oxidation - no absolute numbers => need to combine with flux- and/or emission quantification

Conclusions from the group "Emission measurement methodologies":

General statement:

- There are different goals and contrasting demands:
    1. research versus routine monitoring
    2. accuracy versus costs

- There are different scales:
    1. Whole landfill scale – Plume methods
        - Methane emission is measured
        - To reach oxidation factor, isotopic plume measurement needed
        - high costs, uncertainty when emissions are low and oxidation rate high (not many labs doing isotope analysis)

    2. Whole landfill/sub-area scale - Micrometeorological method
        - CH4 and CO2 fluxes are measured
        - Net "oxidation" derived from these data and landfill gas composition
        - includes also direct emissions and soil respiration
        - To discriminate methane surface oxidation, isotope plume experiments are needed
        - Altogether are expensive
        - Uncertainty unknown

    3. Test cell/bio-window scale
        - Chamber measurements unavoidable
        - discussion on general chamber measurement problems and methodology (diffusion/advection, spatial variability, representativeness…)
        - FID – pattern measurements (qualitative assessment – but can be combined with (chamber/tunnel) flux measurements => e.g., pre-selection tool for chamber placement location)

Conclusions from the group "How to gain reference values for methane load":

Drivers for landfill gas generation – methane flux:

- Concentration gradient: diffusion
- Pressure gradient: advection

Possible methods:

- Concentration gas profile probing/gas well
  - problems: variability of D_{eff} due to water content, soil aggregation
- Carbon balance
  - Uncertainties because of the components of the CO_2 flux
    - CO_2 flux lysimeters in a control setup
- Modelling on LFG production
  - Can we trust the models?
- Waste gas generation potential (e.g., incubation tests in lab)
  - Problem: waste is heterogeneous (composition, moisture)
- Monitor pressure at the base of cover
  - Problems: noise and variability due to wind, local variability due to inhomogeneous water distribution, effect of topography, effect of oxidation
- Combination of istope fractionation and surface CH_4 flux
- Subsurface chamber
  - Preferarably for test cells because of disturbance
- Tracer test: soil gas velocity
  - Covers both diffusion and advection, Tracer has to be similar to CH_4 regarding transport behaviour
- Gas push-pull test
  - Balance reference methane load and emission
  - Picture of activity and kinetics at different depths of cover
  - improve modelling of overall methane oxidation

General statement: Comparison of methods and studies needed!

Assessment of the results, contribution to the future direction of the field:

The key-note presentations and scientific discussions at the workshop showed that currently many research projects are going on related to methane oxidation in landfill covers and engineered systems all over Europe, as well as in the US, Canada, Australia and in Asia. Enhanced methane oxidation in engineered systems on landfills seems to be a powerful tool to reduce greenhouse gas emissions from the waste sector, and the increasing importance and high potential of this measure is evident as it is also mentioned in the 4th IPCC assessment report (2007) as a promising mitigation strategy. During the workshop it becomes obvious that the reliable determination and quantification of the oxidation efficiency is a key-issue in most ongoing research works as well as in practical landfill operation. Landfill operators have to report the annual emission situation of their landfills (impacted also by oxidation processes) under the European Pollutants Release and Transfer Registers (E-PRTR) regulation. At the moment there is no sole consistent, reliable, and comparable solution to address this issue. A lot of different approaches are currently applied, each of these methods has its strengths and limitations with respect to methane oxidation evaluation, and most of the approaches must be modified and adapted for each site specific application to gain reliable results. Many new approaches are under development at the moment but must be advanced and improved, particularly for routine application and cost-efficiency monitoring in the field.

The main findings and musts derived at the workshop are:

- A lot of promising techniques, particularly in the molecular and micro-scale sector are currently under development, but must be specifically advanced for the application on complex landfill samples and in the field;
- Since the advancement and application of field evaluation methods and approaches is very costly and time-consuming, international cooperation and exchange may lead to synergies and more cost-efficient development of new monitoring concepts and methodologies;

Thus,

- a need of harmonization and improvement of existing methods is evident.
- a strong need of international cooperation to pool data and knowledge in order to compare and combine different methods and findings, respectively to test different methods under the same conditions (same landfill site) is obvious.

At the end of the workshop participants envisaged to establish coordinated international research co-operations in the future, preferably within the framework of Research Networking Programmes. Thus, it was considered to draft a proposal to create an international network on this issue.

There was the idea discussed to summarize the scientific output of the workshop in a joint paper to be published in an specific environmental journal, e.g., “Review of Environmental Science and Technology” or “Waste Management”.

Final Programme:

Wednesday, 12 December 2007

15:00 – 18:00 *Arrival to workshop location and Registration*

Session 1:

18:00-19:30

Meeting introduction by the convenors
P. Lechner and M. Huber-Humer (AT)
Institute of Waste Management, BOKU-University Vienna

Presentation of the European Science Foundation (ESF)
Sonja Lojen (ESF Standing Committee for Life, Earth and Environmental Sciences)

Mitigation of Landfill Methane Emissions: Overview from the IPCC 4th Assessment Report introduction of each participant
J. Bogner (US), Coordinating lead author of the 4th IPCC Assessment report, working group III (Chapter 10 - Waste Management))

Round of introduction by all participants

20:00 - *Dinner at the "Heurigen am Reisenberg",get-together and informal discussion*

Thursday, 13 December 2007

Session 2: Microbiological and Lab-scale Approaches:

08:30-09:00

Workshop opening: General survey of various approaches to evaluate methane oxidation efficiency on landfills
M.Huber-Humer and P. Lechner (Austria), Institute of Waste Management, BOKU-University Vienna)

09:00-09:15

DNA- and mRNA-based community analyses of methanotroph populations using a microbial diagnostic microarray
N. Stralis-Pavese (Austria), ARC Seibersdorf Research GmbH, Department of Bioresources/ Microbiology

Questions and sum-up

09:30-09:45

Quantifying the action of methanotrophs in landfills
G. Börjesson (Sweden), Department of Microbiology, SLU-Swedish University of Agricultural Sciences

Questions and sum-up

10:00-10.30 *Coffee break*

10:30-10:45 **Quantification of methanotrophs and evaluation of their activity by quinone and phospholipid fatty acid analysis coupled with a 13C isotopic approach**
A .Watzinger (Austria), Institute of Soil Research, BOKU-University Vienna

Questions and sum-up

11:00-11:15 **Methods to quantify aerobic methane oxidation in landfill covers (activity tests, FISH, DGGE, in-situ microelectrodes)**
P. Lens (Netherlands), Department Environmental Technology, Wageningen University

Questions and sum-up

11:30-11:45 **Standardisation of methane oxidation capacity tests**
J. Gebert (Germany), Institute of Soil Science, University of Hamburg

Questions and sum-up

12:00-13:00 **Group discussion and summary**

13:00-14:00 *Lunch break*

Session 3: Field scale approaches to measure methane emissions and evaluate methane oxidation efficiency

14:00-14:15 **Application of methods to estimate and model methane production, oxidation and emission on landfills**
H. Scharff (Netherlands), Research and development at NV Afvalzorg

Questions and sum-up

14:30-14:45 **New Approaches for Field Quantification of Landfill Methane Oxidation**
J. Bogner (US), Department of Earth and Environmental Science, University of Illinois Chicago

Questions and sum-up

15:00-15:15 **Evaluating the methane oxidation efficiency of a full scale biocover system for greenhouse gas mitigation on a Danish landfill – focus on measuring methane loads**
P. Kjeldsen (Denmark), Institute of Environment & Resources, Technical University of Denmark

Questions and sum-up

15:30-15:45 **Methane and Carbon Dioxide Emission Measurements using the Micrometeorological Eddy-covariance Method**
T. Laurila (Finland), Finnish Meteorological Institute, Climate and Global Change Research

Questions and sum-up

16:00-16.30 *Coffee break*

16.30-16:45	**Assessing methane oxidation efficiency in biocovers –combination of different approaches in an Austrian case study** **M. Huber-Humer (Austria)**, Institute of Waste Management, BOKU-University Vienna
	Questions and sum-up
17.00-17:15	**New approach to measure in-situ methanotrophic activity in the field** **J. Gebert (Germany)**, Institute of Soil Science, University of Hamburg
	Questions and sum-up
17.30-17:45	**Stable isotope method and modelling to quantify methane oxidation** **K. Mahieu (Belgium - current post doc in US)**, Department of Applied Analytical and Physical Chemistry, Ghent University
17.45-18.00	**Stable isotope approach applied on landfill biocovers** **Ch. Scheutz (Denmark),** Institute of Environment & Resources, Technical University of Denmark
	Questions and sum-up
18.15-19:00	**Group discussion and summary**
19:15-20:15	*Possibility to visit one of the famous Viennese "Christmas Markets"*
20:15-	*Dinner at the brewery "Schlossbräu"*

Friday, 14 December 2007

Session 4: Interactive group discussion and draft of outcome report:

08.30-09:00	**Wrap up and formation of small discussion groups** Topic: Draft of a standard procedure for methane oxidation evaluation on landfills
09.00-10:30	**Small group discussion and draft of group reports**
10:30-11:00	*Coffee break*
11.00-13:00	**Short presentation of small group reports, joint discussion and outlook on future processing and cooperation**
13:00–14.00	*Workshop closing and lunch*

Statistical information on participants:

(without ESF-representative)

22 persons attended the workshop coming from 10 different ESF-member countries and two overseas countries.

Gender participation

Total participants

Female	10	(45.5 %)
Male	12	(54.5 %)

Key-note speakers

Female	6
Male	7

Nationality

Austria	5
Belgium	1
Canada	1
Denmark	2
Finland	1
France	1
Germany	4
Italy	1
Netherlands	2
Poland	2
Sweden	1
United States	1

Age distribution – stage of profession:

1) < 30 years	ca. 14%
2) 30 – 40 years	ca. 41%
3) 40 – 55 years	ca. 31%
4) >55 years	ca. 14%

I) PhD-Students, junior researcher,…	3	(14%)
II) PostDoc, Research Assistant, Assistant Professor,…	9	(41%)
III) Associate Professor, Senior Researcher, Senior Manager,…	8	(36%)
IV) Full Professor	2	(9%)

Final List of Participants:

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