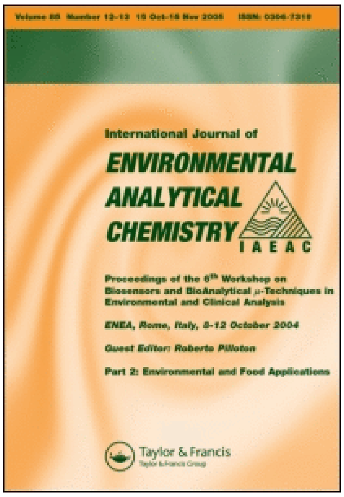

International Journal of Environmental Analytical Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713640455

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To cite this Article Öman, Cecilia(1998) 'An Automated Experimental Equipment for Studying the Fate of Organic Compounds in Landfills', International Journal of Environmental Analytical Chemistry, 71: 1, 73 — 85

To link to this Article: DOI: 10.1080/03067319808032618

URL: http://dx.doi.org/10.1080/03067319808032618

Intern. J. Environ. Anal. Chem., Vol. 71(1), pp. 73-85
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AN AUTOMATED EXPERIMENTAL EQUIPMENT FOR STUDYING THE FATE OF ORGANIC COMPOUNDS IN LANDFILLS

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(Received 20 March 1997; In final form 14 August 1997)

An experimental system for simulating and analysing the fate of organic compounds in landfill-like environments has been developed. The performance of the system was investigated by using 11 volatile organic compounds which have previously been identified in landfill leachates. The compounds represented a range of hydrophobicity and the limits of quantification were at µg/l-levels similar to that in actual landfill leachates. The automatised system consisted of a reservoir without headspace for storing the water solutions of the volatile test compounds, a reactor for studying the landfill processes, a pump, a Purge and Trap concentrator, a gas chromatograph (equipped with flame ionisation and electron capture detectors), and a computer for data collection.

Keywords: Landfill; fate; organic compounds; sorption; Purge and Trap concentrator; gas chromatography

INTRODUCTION

Hazardous organic compounds have been identified in landfill leachates and landfill gases. The tendency of specific compounds, released from the waste, to be emitted from landfills is related to their transformation, evaporation and sorption to the waste. These processes can be described by the physical, chemical and biological properties of the compounds and of the landfills [1]. Sorption, or partition, of the organic compounds to the waste affects the transport rate of compounds through the landfill and thus the residence time. In contrast to studies focusing on sorption to soils and sediments [2–4] only a few investigations on sorption of organic compounds to waste have previously been published [5]. Dobbs et al. [6] studied sorption to waste water solids.

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Results from investigations of groundwater at the vicinity of waste disposal sites in Germany and in the U.S.A. [7], and from literature reviews on investigations of landfill leachates [8], show a predominance of volatile organic compounds. Christensen et al. [9] and Öman [8] conclude that specific organic compounds, excluding the volatile fatty acids during the acid phase, often are present in landfill leachates in concentrations at µg/l levels.

The purpose of this study was to develop an experimental method to enable investigations of the processes in landfills, such as sorption/desorption and transformation of specific organic compounds, under realistic conditions. The method was developed to; i) study volatile organic compounds which have frequently been found in actual landfill leachates, ii) study the compounds at the low µg/l, occurring in landfill leachates, and iii) to be less time-consuming than experimental batchwise methods.

MATERIALS AND METHODS

Equipment

Grice et al. [10] presented a continuous flow method for studying the sorption of organic compounds to soil. This method was later modified by Williamson et al. [11]. The continuous flow methods proved to be less time consuming than the traditionally used batch method and produced isotherms with higher degrees of resolution. Both methods originally used spectrophotometers for the analysis of components in the aqueous phase. Developmental progress has however led to replacing the spectrophotometer with a Purge and Trap concentrator (PAT) coupled to a gas chromatograph (GC). By purging the water samples before injection in the GC the PAT system facilitates automatisation. The analytical procedure increases the number of volatile organic compounds that can be analysed at concentrations previously found in landfill leachates i.e. µg/l levels. In addition, the use of a GC coupled to a PAT instead of using a spectrophotometer reduces the limits of quantification for the compounds to be analysed. This is particularly true for analysis of organochlorine compounds using an electron capture detector (ECD).

The experimental equipment consisted of a vessel for storing the test solution, a reactor for studying the landfill processes, a dosimeter for sampling, and a Purge and Trap concentrator coupled to a gas chromatograph for analysis (Figure 1). Data were collected by a computer and the system was automated using a Varian Star data system. All experiments were run at room temperature.

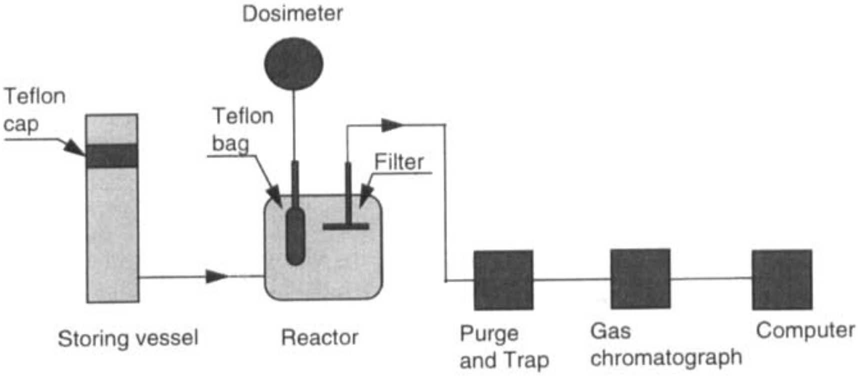

FIGURE 1 The experimental equipment. The landfill processes are simulated in the reactor

Only glass, stainless steel or PTFE (polytetrafluoroethylene, Teflon) components were used. Prior to use all materials were washed with detergent and rinsed with tap and then with organic free water. The apparatus was placed in an oven at 105°C with air ventilation for one hour. Components with intricate and fine parts were rinsed in ultrasonic baths, and then with organic free water before they were exposed to 105°C with air ventilation for one hour. The glass filters were soaked in dichromate sulphuric acid for four days, and then with organic free water before heating to 105°C with air ventilation for one hour.

Storage vessel

Water solutions of the organic compounds were stored in a glass cylinder, equipped with a PTFE cap floating on the surface of the water solution. The PTFE cap prevented volatilisation of organic compounds during the experiments. The vessel had a precise inner diameter of 50 mm and the PTFE cap was designed to leave a minimum of space between the cap and the glass wall. Water was added between the cap and glass wall leaving no air space for volatilisation of the compounds. The water solutions were continually stirred during the experiments. Unfortunately, the PTFE cap was found to release organic analytes after months of contact with water solutions of the compounds. Contaminated caps were thus replaced periodically.

Reactor

The reactor was continually stirred and consisted of a 1.0 dm^3 glass container supplied with a glass filter disc in front of the outlet. The reactor was also supplied with a PTFE bag coupled to an automatised dosimeter (Metrohm, Dosimat

725). The PTFE bag was filled and emptied by the dosimeter creating a flux of the aqueous phase from the storage vessel, passing through the reactor, to the analysing equipment. This enabled samples to be taken after equilibrium had been reached but before the introduction of new test solution. The direction of the water flux was controlled by check valves.

Filter

The distinction between the solid phase and the water phase had to be defined operationally by filtration. It was found that filter pore sizes smaller than 9–15 μm could not be used due to the resulting backpressure. When filtering the solid waste through a 9–15 μm pore size filter 3 dry weight percent of the solids passed through the filter. In order to eliminate excessive solid waste particles in the filtrate, the waste was pre-washed before use in the experimental tests through a 9–15 μm pore size filter until no waste particles could be measured in the filtrate. Due to the continuous stirring of the waste in the reactor during the experimental tests however attrition of the solid waste occurred which generated solids in the filtrate after some time. In practise the amount of solid waste in the filtrate after 24 hours stirring of pre-washed waste was determined as 3 dry weight percent (triplicate measurements).

Purge and Trap concentrator (PAT)

The Purge and Trap concentrator (Tekmar LSC-3000) purges the volatile compounds from an aqueous sample by bubbling helium gas through the sample. The purged compounds are subsequently trapped in a tube containing a sorbent material (Tenax, 2,6-diphenylene oxide polymer). When purging was completed, the sorbent tube was back flushed with helium at 180°C to desorb the trapped compounds onto a capillary gas chromatography column for separation and analysis.

Each day prior to use, the trap was conditioned for 20 min at 220°C with flush in the "bake mode" and then for 20 min at 225°C with back flush in the "desorb mode". During conditioning under the desorb mode the trap was vented to the gas chromatographic column. The column was then conditioned at 200 °C for 20 min.

On delivery from the manufacturers, the vessel used for purging the water samples was pressurised to coincide with sample injection. This pressure was found however to inhibit the injection by the dosimeter. Therefore, a pressure equilibration valve was installed after the purge vessel and the high pressure was reduced just before the time for injection. This valve was then kept closed during the injection causing no losses from evaporation of the organic compound analytes.

Gas chromatograph (GC)

The gas chromatograph used in the system was a Varian 3400 equipped with a 60 m long × 0.75 mm ID VOCOL (Supelco Inc.) wide-bore capillary column, with a 1.5 μm film thickness. The column was kept at 30°C for 8 minutes, after which, the temperature was programmed to rise to 180°C at 4°C/min, where it was maintained for 10 min. The flow rate of helium carrier gas was adjusted to about 9 ml/min. The column was connected directly to the end of the transfer line from the Purge and Trap concentrator, and was run with cryogenic cooling using carbon dioxide for focusing the analytes before increasing the temperature. Two detectors were connected in parallel, a flame ionisation detector (FID) and a electronic capture detector (ECD), by splitting the carrier gas using a pressfit. The flow through the ECD was reduced to half the flow through the FID by using different lengths of the capillary tube from the pressfit to the detectors. The detectors were held at 200°C.

Chemicals

The performance of the equipment was evaluated using 11 organic compounds previously found in landfill leachates. The compounds were chosen to represent a range of hydrophobicity measured as the octanol/water partition coefficient, K_{ow} (Table I).

All chemicals were of analytical quality and were purchased from the following suppliers; methanol, dichloromethane, trichloromethane, trichloroethene, benzene and toluene from Merck (Darmstadt, Germany), ethylbenzene, isopropylbenzene, 1,4-dichlorobenzene, 1,2,4-trichlorobenzene, from Merck (Schuchart, Munich), hexachloro-1,3-butadiene from Aldich-Chemie (Steinheim, Germany), 1,2-dichloroethane from BDH (Poole, England) and naphthalene from Fisher Chemicals (New Jersey, U.S.A.).

The water used was treated by reverse osmosis, ion exchange and then by a Milli-Q system. The Milli-Q water was further checked to be free of analytes. Stock standard solutions of the analytes were prepared in methanol and subsequently diluted with Milli-Q water to provide the concentrations as desired. Analyte solutions with concentrations above one tenth of the water solubility for the most hydrophobic compound, were mixed for 24 hours before use. The samples were run immediately or directly after mixing.

All water solutions used in the experiments had been buffered to pH 7 with sodium hydrogen phosphate and sodium dihydrogen phosphate. The water solutions also included 0.2 weight percent sodium azide in order to prevent microbial degradation of the waste.

TABLE I The compounds included in this study together with their log K_{ow}values. Precision of repetitive measurements with the PAT-GC separately, and with the PAT-GC coupled to the reactor and the storing vessel have been compiled. Limits of quantification for the PAT-GC coupled to the reactor and the storing vessel were compared with previous results from studies with PAT and GC, separately [16]. All units were in µg/l except the relative population standard deviations (RSD) which were in percent.

Compound	*CAS Registry Number*	*log K_{ow}*	*Precision. RSD, %*		*Limit of quantification*	
			PAT-GC only	*PAT-GC and glassware*	*PAT-GC and glassware*	*PAT-GC separately [16]*
Dichloromethane (methylene chloride)	75–09–2	1.2	1.5	7.2	1.9	0.6
Trichloromethane (chloroform)	67–66–3	2.0	2.6	4.5	0.9	1.5
Benzene	71–43–2	2.0	1.6	2.8	0.7	0.2
Trichloroethene	79–01–6	2.3	1.4	2.0	0.2	0.6
Toluene	108–88–3	2.6	2.9	5.1	1.2	0.2
Ethylbenzene	100–41–4	3.2	1.2	2.3	0.4	7.3
Naphthalene	91–20–3	3.3	2.1	1.6	0.2	
1,4-Dichlorobenzene	106–46–7	3.4	1.3	2.7	0.3	0.2
Isopropylbenzene (cumene)	98–82–8	3.7	1.1	3.4	0.4	
1,2,4-Trichlorobenzene	120–82–1	4.2	1.2	3.0	0.4	
Hexachloro-1,3-butadiene	87–68–3	4.8	3.8	4.2	0.6	

Log K_{ow} are averages of measurements in octanol from Hansch and Leo [28] except 1,2,4-trichlorobenzene and hexachloro-1,3-butadiene which are from the Swedish National Chemicals Inspectorate [29].

RESULTS AND DISCUSSION

Compound losses during experimental runs

Precautions

Photodegradation was eliminated by wrapping the glassware containing water solutions in aluminium foil. Catalysis of hydrolysis reactions by hydrogen ions or hydroxyl ions [12] were minimised by buffering the system to pH 7 with sodium hydrogen phosphate and sodium dihydrogen phosphate. A number of substances (dichloromethane, benzene, toluene, ethylbenzene and naphthalene) are reported not to be abiotically hydrolysed under neutral pH-values. Trichloromethane has, however, been shown to be hydrolysed but only to 0,1 percent during 24 hours [12] which was considered negligible. Microbiological transformation was suppressed by adding 0.2 weight percent of sodium azide, NaN_3, to all water solutions. The reductive transformations of the halogenated hydrocarbons was suppressed in the present system, as the redox potential as was kept above 50 mV (relative to Ag:AgCl) at all times [13]. Evaporation was minimised by eliminating headspace in the reactor and by using the floating PTFE cap in the storage vessel. Adsorption to surfaces was minimised by using borosilicate glass, PTFE, or stainless steel for all surfaces in contact with the aqueous solution. With these preventive measures used in the system, the actual losses were experimentally determined for all compounds.

Storage vessel

Significant losses were measured while pouring water solutions containing the analytes, sodium azide and pH-buffer into the storage vessel and into the reactor. The losses were due to evaporation and to adsorption to the walls. This was corrected for in the storage vessel by determining the concentrations of the analytes after pouring the water solutions into the vessel and after closing the vessel with the PTFE cap. Actual losses in the storage vessel during 24 hours were then found to be less than 3 percent, indicating no significant further evaporation or adsorption to the walls of the storage vessel, and no significant abiotic hydrolysis, redox transformations, biological transformation or photodegradation in the water solutions. The analyses were made in triplicate at analyte concentrations ranging between 200 and 1700 μg/l (representative of those solutions in the storage vessel during experiments with waste in the reactor).

Reactor

The losses of analytes from the water solution in the reactor during 24 hours were found to be significant. The losses varied between 4.8 and 30 percent with an average of 12 percent. If the PTFE components were excluded from the reactor the losses over this period were reduced to the range 0.0 – 11 percent with an average of 4.5 percent. Besides the losses attributed to adsorption to the PTFE components, adsorption to the glass surfaces in the reactor could also be assumed. These assumptions were supported by the fact that the degree of losses increased with the hydrophobicity of the compounds tested.

The losses in the reactor described above however were determined in clean systems with the analytes solved in Milli-Q water. It could be assumed that if the reactor also contained waste, which would always be the case during actual experiments, the adsorption of the analytes to waste would dominate and thus adsorption to the PTFE components and glass surfaces could be considered negligible. In order to verify this, 1 gram of pre-washed solid waste was equilibrated with the analytes, sodium azide and pH-buffer during 24 h in the reactor. The waste was then soxhlet extracted with methanol giving recoveries from 42 to 105 weight percent. The degree of recoveries were directly correlated to the volatility of the compounds, with the lowest recoveries for the most volatile compounds. Thus the low recoveries were assumed to be due to the open handling of water solutions and waste, and not due to adsorption to the PTFE or glass materials used. The compounds tested had Henry's law constants ranging from 0.005 to 2.30 kPa m^3/mole [14]. With exception of the four most volatile compounds, the recoveries ranged between 91 and 105 percent which indicated negligible adsorption to the inside of the reactor when waste was present.

A further investigation was conducted for the eight most hydrophobic compounds by extracting the inside of the reactor with methanol after the reactor had contained water solutions of organic compounds, with and without the addition of solid waste. The water solution contained about 300 µg/l of each compound. Significant losses due to evaporation could be expected. The extraction showed that 6.2 – 14 percent of the initial concentrations could be extracted from the reactor walls when the reactor did not contain waste while less than 1.7 percent of the analytes could be recovered with waste present.

For comparison it can be noted that Grathwohl [4] observed severe losses of trichloroethane and other organic compounds from blank sample vials, due to adsorption or permeation through PTFE-lined rubber septa. When a sediment containing 30 percent organic carbon was present in the same vials, however 98.55 percent of the trichloromethane was sorbed to the sediment, 1.19 percent was dissolved in the water, 0.19 percent was evaporated and a negligible 0.06 percent was lost. Thus, the losses of trichloromethane were reduced from severe to negligible when the sample in the system was changed from pure water to a mixture of water and sediment.

Filter

Severe losses were also found with previously exposed filters, probably due to sorption to waste residues trapped in the filter material. These losses were eliminated by soaking the filters in dichromate sulphuric acid for 4 days.

Compounds separation

Benzene and 1,2-dichloroethane could not be properly separated by the GC column used despite careful optimisation of the GC and PAT programs. The detector responses of the two components were not additive, thus the benzene response could not be calculated by subtracting the 1,2-dichloroethane response measured by the ECD.

TABLE II Previously quantified levels in leachates from actual landfills of the compounds included in this study (references given in parentheses). All units were µg/l. Ranking figures based on the detection frequency have also been compiled.

Compound	*Median*		*Range or individual sample*									*Rank*
	(7)	*(18)*	*(19)*	*(20)*	*(21)*	*(22)*	*(23)*	*(24)*	*(25)*	*(26)*	*(27)*	*(7)*
Dichloromethane		483	15	17000	6.0	1.1–84						
Trichloromethane	2.7	7	30–70		6.6–8.5	<0.1–0.8	21800					12
Benzene	11	11 100	4–31	3800	1.1–572		9550	2.1–4.7	1–8			6
Trichloroethene	3.5	19		360	2.3–7.9		2140	<0.01–5.3	0.05–0.44			4
Toluene			2399-7000	9400			660000	0.21–140	21–500			2
Ethylbenzene		44		2200	1.7–20	<0.5-1400		≤480	6–100			10
Naphthalene		34		290				3–110	2–18			11
1,4-Dichlorobenzene		14						7–40		<1-1.1		
Isopropylbenzene		1										13
1,2,4-Trichlorobenzene											ID	
Hexachloro-1,3-butadiene											ID	

7) The medians, and the ranking figures based on the detection frequency, of 15 main contaminants found in the groundwater downstream of 92 waste deposit sites in Germany.
18) The medians of compounds found in leachate from 56 landfills in Wisconsin.
ID=Identified but not quantified.

Solid waste

The solid waste had been collected from a separation plant (Brini classifier MK VIII) at the municipal landfill of Kovik in Sweden. The fraction used, the light fraction, had been separated from metal, rubber, solid plastic, food residues, sand and glass. The light fraction represents 50–70 volume percent of the waste in Sweden and consists of paper (50 percent), plastics (25 percent), textiles, organic package materials, wood and inorganic waste. The fraction was determined to contain 47 weight percent organic. carbon (average values from three air-dried samples). The waste was dried at 70°C until stable weight was reached and then shredded to a particle size of less than 10 mm (Moco, shredding machine AZ 7). Before the experimental tests, the waste was pre-washed in order to reduce the amount of waste particles in the filtrate. Pre-washing was performed by mixing the waste with organic free water and filtering the mixture through an identical filter as that used during the experimental trials. This procedure was repeated until the filtrate was clear and thus contained reduced amounts of waste particles. Finally, the waste was air dried and kept dry until use.

Microbial stability

In order to determine if microbial degradation of the waste could be suppressed by sodium azide, a water solution of 0.2 weight percent sodium azide was prepared. Solid waste was added and the mixture was stirred for 36 hours, while the redox potential and the pH was measured continuously. The results showed stable redox potentials and stable pH-values during the 36 hours. In a parallel study where the sodium azide was excluded, the redox potential was reduced from 160 mV to minus 210 mV (relative Ag:AgCl), while the pH decreased from 6.5 to 5.9 during the same period of time. Thus it was concluded that microbial degradation of the solid waste in the reactor could be neglected when 0.2 weight percent sodium azide was present in the sample solution.

EQUIPMENT EVALUATION

Precision

The precision of the PAT and GC was determined using Milli-Q waters fortified with organic compounds at concentrations between 0.4 and 3.4 μg/l according to Ho [15]. Five samples were measured consecutively from the same water solution during one day (repetitive measurements). In addition, five replicate samples were measured from five separately prepared water solutions during the next day (replicate measurements). The obtained results from all ten samples gave a precision in the range of 5.3 % to 9.8 % relative population standard deviation (RSD) with an average of 7.7 %. The RSD from the five repetitive measurements were in the range of 1.1 % to 3.8 %, with an average of 1.9 % (Table I) and the RSD from the following five replicates were in the range of 1.2 % to 6.5 % with an average of 2.8 %.

The precisions associated with the PAT and GC coupled to the rest of the glassware equipment were determined by five further repetitive measurements. The reactor was coupled to the storage vessel and the samples were injected using the dosimeter, as during actual experimental runs. Water samples were injected with one hour intervals and the determined RSD ranged from 1.6 % to 7.2 % with an average of 3.5% (Table I).

Sensitivity

The sensitivity of the method was determined from water samples spiked with the organic compounds, and expressed in terms of the limit of quantification (LOQ). LOQ was defined as 10 times the population standard deviations (SD) above the measured average blank value of five Milli-Q water blanks. If the compounds were not found in the blanks, the limits of quantification were determined as 10 SD above the noise level according to Westendorf [16] and Sporstøl [17]. LOQ for the PAT-GC using the FID detector varied between 0.11 µg/l and 0.96 µg/l with an average of 0.39 µg/l. LOQ for the PAT-GC coupled to the glassware equipment varied between 0.21 µg/l and 1.94 µg/l with an average of 0.66 µg/l (Table I). The determined limits of quantification were similar to results previously presented by Westendorf [16] where PAT-GC had been used separately.

Quantified levels of the organic compounds in actual landfill leachates have been reported to lie between µg/l levels to mg/l levels. Previous results compiled in Table II indicate that organic compounds are primarily present at µg/l levels in landfills and that in exceptional cases, strong leachates have been sampled with relatively high concentrations at mg/l .

APPLICATION TO LANDFILL PROCESSES

In the reactor different landfill processes can be simulated such as sorption and desorption of organic compounds to solid waste and biological transformation of organic compounds in the presence of solid waste. If sorption – desorption processes are to be studied the system can be operated so that biological transformations can be suppressed. If attention is to be focused on microbial activity however, sorption – desorption processes and biological transformations can be studied simultaneously.

When using the equipment for sorption studies (Öman and spännar, submitted), the experiments begun with the reactor containing solid waste mixed in water solutions of sodium azide and pH-buffer, without the organic compounds. At pre-selected intervals samples were transferred from the reactor and replaced with equal amounts of a second buffered water solution kept in the storage vessel containing the organic compounds to be studied. Each time the water samples from the storage vessel were introduced to the reactor, the amount of the organic compounds in the reactor increased. The sorption isotherm could then be constructed using a simple mass balance, i.e.; mass sorbed = mass inflow – mass outflow – mass in solution.

CONCLUSIONS

The described experimental equipment was shown to be suitable for studying the fate of volatile organic compounds in simulated landfill environments at actual concentration levels i. e. µg/l. The automatisation associated with the system decreased the time needed for the generation of sorption isotherms compared to conventional batch experiments. The precision for repeated measurements was below 7 percent (relative population standard deviation).

When studying hydrophobic volatile organic compounds in water at µg/l levels, the losses of compounds due to sorption to the equipment, evaporation or transformation during storage and experiments may be severe and precautions are necessary. In the described apparatus these compound losses were reduced to negligible levels.

Acknowledgements

The author is grateful to Camilla Spännar and Anna Björklund for laboratory assistance, and to Christer Permér for development of the specially designed PTFE components. The project was funded by the Swedish Waste Research Council and the Swedish Environmental Research Institute.

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