Methodology for evaluation of hazards from solid waste and landfill generated leachate

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Methodology for Evaluation of Hazards from Solid Waste and Landfill-Generated Leachate

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Centre for Aquatic Biology and Chemistry

2008

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Abstract

A methodology based on an analytical protocol for evaluation of hazards from landfill leachate and solid waste is described. A dynamic analytical protocol, the LAQUA protocol, including measurements of inorganic and water-quality parameters, polar and non-polar organic marker compounds, and toxicity, was constructed. An acute toxicity test, using the brackish water crustacean Artemia salina as test organism, was developed. The methodology was applied to authentic problems such as investigation of different treatment techniques for landfill leachate, evaluation of leaching tests and characterization of solid wastes, and an investigation of a filter material aimed for leachate treatment. Investigated treatment methods comprised in all cases pre-treatment by aeration combined with sedimentation, followed by one of: bioremediation, ozonation, chemical oxidation by Fenton's reagent, or geo-bed filters. Evaluated filter materials were mixtures of natural or residual waste products. A combination of pre-treatment followed by a geo-bed filter containing a mixture of peat and carbon-containing ash gave an efficient simultaneous removal of metals and organic pollutants. The performance of two leaching tests for characterization of solid waste, the up-flow percolation test (SIS-CEN/TS 14405:2004) and the batch test (SS-EN 12457 -3), was investigated. Solid waste materials (sludge from street gutters and fragmented metallic waste) were characterized using leaching tests and the hazards of the materials were evaluated in the eluate, obtained at specific liquid-to-solid ratios (L/S). The L/S 2 and L/S 10 values were compared with limit values included in the waste acceptance criteria (WAC). The analyses were extended towards specific organic compounds, such as individual phenolic compounds and polychlorinated biphenyls (PCB). Organic compounds were found in eluates from both types of tests, showing the possibility to use these methods to evaluate the leaching of such compounds from waste materials. The use of authentic leachate as leachant, leads to increased concentrations of heavy metals in the eluate, compared to the prescribed use of demineralised water as leachant. Generally good agreement was found between the results of the two leaching methods. A strategy based on batch tests is described for investigation of a filter material for leachate treatment. Batch tests gave suitable information about the leaching from new and used material, and showed high removal efficiencies of metals and non-polar organic compounds. However, for investigation of removal of polar organic markers (e.g. phenolic compounds) a batch test is not sufficient and should be supplemented by a column test.

Key words: Analytical protocol, organic pollutants, toxicity test, landfill leachate, leachate treatment, leaching tests, waste acceptance criteria

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The thesis is based on the following papers, referred to in the text by their roman numerals:

I. **Development and application of an analytical protocol for evaluation of treatment processes for landfill leachates. I. Development of an analytical protocol for handling organic compounds in complex leachate samples**
Bergström, S., Svensson, B.-M., Mårtensson, L. and Mathiasson, L.
International Journal of Environmental and Analytical Chemistry, **2007**, *Vol. 87, No. 1*, 1-15

II. **Development and application of an analytical protocol for evaluation of treatment processes for landfill leachates. II. Evaluation of leachate treatment efficiency of different steps in a constructed pilot plant**
Mårtensson, L., Bergström, S., Svensson, B.-M. and Mathiasson, L.
International Journal of Environmental and Analytical Chemistry, **2007**, *Vol. 87, No. 1*, 17-27

III. ***Artemia salina* as test organism for assessment of acute toxicity of leachate water from landfills**
Svensson, B.-M., Mathiasson, L., Mårtensson, L. and Bergström, S
Environmental Monitoring and Assessment, **2005**, *102*, 309-321

IV. **Leachability testing of metallic wastes**
Svensson, B.-M., Mårtensson, L., Mathiasson, L. and Eskilsson, L.
Waste Management and Research, **2005**, *23*, 457-467

V. **Leachability testing of sludge from street gullies**
Svensson, B.-M., Mårtensson, L., Mathiasson, L., Åkeson, T. and Persson, A.
Waste Management and Research, **2006**, *24*, 260-268

VI. **Column studies aiming at identification of suitable filter materials for pollutant removal from landfill leachate**
Kängsepp, P., Svensson, B.-M., Mårtensson, L., Rosenquist, D., Hogland, W. and Mathiasson, L.
International Journal of Environment and Waste Management, **2008,** *2 (3/4)***,** In press

VII. **A batch-test-based approach towards an environmental friendly adsorbent for leachate treatment of organic and inorganic pollutants**
Kängsepp, P., Svensson, B.-M., Mårtensson, L. and Mathiasson, L.
Manuscript, submitted to *Journal of Environmental Management,* **2008**

Contribution by the author to the different papers

Paper I. The author was involved in the development of the LAQUA protocol and was involved in the scientific discussion during the preparation of the manuscript.

Paper II. The author was involved in the design of the pilot plant and made a substantial part of the sampling strategy. The author performed a part the measurements and was involved in the scientific discussions during the preparation of the manuscript.

Paper III. The author designed and performed a major part of the experimental work and wrote the major part of the paper.

Paper IV and Paper V. In these papers, the author was closely involved in the experimental design, performed a substantial part of the experiments and the evaluation of the results. The author wrote the major part of the papers.

Paper VI. The author was involved in the experimental design and made a substantial part of the sampling and measurements. The author was involved in preparation of the paper.

Paper VII. The author was highly involved in the experimental design and performed a part of the experiments and the data evaluation. The author contributed actively to the scientific discussions and the writing of the manuscript.

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Populärvetenskaplig sammanfattning

Avfall har alltid uppkommit i alla samhällen i alla tider. Det vanligaste sättet att hand om det har varit att samla avfallet i soptippar (deponier) på mark som ansetts obrukbar, t.ex. i sankmarker utanför bebyggelsen. I takt med ökad konsumtion och produktion har också mängderna avfall ökat. Anledningen till att avfall och avfallshantering kommit alltmer i fokus i miljödebatter under de senaste decennierna, är inte bara de stora mängderna avfall på de många och stora sopbergen runt om i världen. Deponierna släpper också ifrån sig miljöfarliga ämnen från de alltmer komplexa produkter som blivit lagda i deponin. Dessa kommer att fortsätta att läcka ut länge efter det att deponeringen på soptippen slutat, och skapa problem för människor och miljö i flera generationer.

Som en konsekvens av internationella beslut bl.a. Agenda 21 (FNs miljökonferens i Rio) och EUs lagstiftning om avfallshantering som Sverige har tagit in i sin lagstiftning, kommer bl.a. antalet soptippar att minska. Efter den 31 december 2008 beräknas endast ett 90-tal deponier för kommunalt avfall att vara i drift i Sverige. Lagstiftningen beskriver en avfalls hierarki där deponering är den sämsta och absolut sista åtgärden som får göras bara när inget av de andra alternativen kan uppfyllas. Det ställs stora krav på de soptippar som är godkända enligt den nuvarande lagstiftningen. Olika typer av deponier ska finnas för olika slag av avfall. En deponi för farligt avfall har bättre skydd mot läckage både under och över tippen än en tipp för icke-farligt avfall. För att ha kontroll på vilket avfall som läggs på deponin och därmed kunna behandla det på korrekt sätt, måste avfallet beskrivas och klassas innan det skickas för deponering. Karakteriseringen görs bl.a. med hjälp av lakningstester.

Syftet med denna avhandling har varit att utarbeta en metodik för at utvärdera miljöfarliga ämnen som kommer från avfall och lakvatten från soptippar. Den inledande forskningen utgjordes av ett projekt kallat Laqua, finansierat av EU-kommissionens program för samarbete inom Östersjöregionen, SweBaltcop. Projektets uppgift var att främja utveckling av ekologiskt och ekonomiskt hållbara reningsmetoder för lakvatten.

Lakvattnet bildas främst av nederbörd som faller över deponin. Det vatten som kommer in i soptippen tar med sig många av de ämnen som finns i tippen när det rinner ut. Dessa ämnen kan komma från sådant som har deponerats eller bildas när avfallet bryts ner. Lakvattnet

samlas upp och renas på något sätt innan det släpps ut till ett naturligt vattendrag. En vanlig metod att behandla lakvatten är att pumpa det till det kommunala avloppsreningsverket och rena det tillsammans med avloppsvatten. Men detta är ingen optimal lösning eftersom lakvatten innehåller andra föroreningar än avloppsvatten, t ex salter, tungmetaller och svårnedbrytbara organiska föreningar. Avloppsreningsverken är konstruerade för att rena avloppsvatten och lakvattnets föroreningar kan störa reningsprocessen. Framför allt kan de känsliga mikroorganismerna i det biologiska reningssteget påverkas negativt. Slammet som bildas vid reningsprocessen kommer att koncentrera många av de oönskade föroreningarna som härstammar från deponin. Slammet är egentligen ett utmärkt gödselmedel för jordbruket, men på grund av att slammet är förorenat av tungmetaller och svårnedbrytbara organiska ämnen kan inte slammet användas. Slammet blir då ett avfall som kommunen inte kan bli av med utan stora kostnader. Många kommuner har därför valt en separat rening av lakvattnet. Val av reningsteknik är beroende av flera faktorer, som volymerna lakvatten som uppkommer, innehållet av de olika miljöfarliga ämnena, vart det renande vattnet ska släppas ut och utrymme för att bygga en reningsanläggning.

För att utvärdera olika tekniker för rening av lakvatten, byggdes en försöksanläggning på soptippen i Kristianstad. För att kunna utvärdera en reningsmetod måste bestämningar av koncentrationer av olika ämnen (analyser) göras. På grund av den ökande oron för organiska miljögifter som PCB och fenoler, skulle reningsteknikerna utvärderas med fokus sådana eller liknande ämnen. Analyser av organiska ämnen är komplicerade och tidskrävande, och det är inte möjligt att analysera alla ämnen. I många undersökningar används endast generella parametrar för att uppskatta innehållet av organiska ämnen, men dessa metoder ger ofta inte tillräcklig information om det egentliga innehållet i lakvattnet. Därför utarbetades ett utvärderings protokoll, LAQUA protokollet (artikel I) för bestämning av organiska miljögifter i olika förorenade vatten. Detta protokoll innehåller förutom analyser för organiska miljögifter som PCB och fenoler och en akut toxicitetstest, även standardiserade rutinanalyser av metaller och vattenkemiska parametrar. Eftersom separat analys av alla organiska ämnen inte nödvändig för att bedöma olika reningsmetoder för lakvatten, innehåller protokollet ett antal markörer för polära, respektive opolära organiska föreningar.

Den biologiska giftighetstesten som utvecklades (artikel III) och ingår i protokollet är ett s.k. akut toxicitetstest, dvs. organismen påverkas direkt av höga halter av föroreningar. I testen används det lilla saltvattentåliga kräftdjuret *Artemia salina*, som säljs som föda åt akvariefiskar. En bestämd volym med ett antal *Artemia* larver läggs i små brunnar med olika koncentrationer av lakvatten under 24 timmar. Sedan jämförs vid vilken inblandning av lakvatten som hälften av kräftdjuren har fått rörelsestörningar. Resultaten på tester med obehandlat lakvatten, respektive behandlat med olika reningstekniker jämförs, och på det viset kan effektiviteten på reningsmetoder bedömas gentemot en vattenlevande organism.

Utvärderingen av försöksanläggningen (artikel II) visade att vid förbehandlingen, bestående av luftning och sedimentering, togs mycket av föroreningarna bort, och det rekommenderas att ett sådan reningssteg alltid bör finnas vid en reningsanläggning för lakvatten. De kemiska behandlingsmetoderna med ozon och Fentons reagens (tvåvärt järn och väteperoxid) var effektiva på att ta bort de organiska miljögifterna, men även de mer kostnadseffektiva filterbäddarna visade sig fungera bra.

Den goda erfarenheten från försöksanläggningen av filterbäddar ledde till att effektiviteten hos fler filter material undersöktes. I artikel VI beskrivs ett försök, gjord i laboratorium, där lakvatten från en soptipp som tar hand om industri avfall (metallavfall från bl.a. bilar och kylskåp) fick rinna genom kolonner med olika filtermaterial. Mixen av torv och aska med kolinnehåll visade sig vara bra på att ta bort både metaller och organiska ämnen från lakvattnet. Kunskapen från bl.a. dessa undersökningar har bidragit till att en fullskaleanläggning för lokal rening av lakvatten har kunnat byggas i anslutning till Stena metalls soptipp i Halmstad.

Den andra delen i avhandlingsarbetet riktade in sig mot lakningstester. För att undersöka vilka ämnen som kan lakas ut från ett avfall rekommenderas två olika standardiserade lakningstests metoder. Vid den ena metoden pumpas en vätska genom en kolonn med en uppvägd mängd avfall tills ett visst vätske/fast fas förhållande (L/S halt) har uppnåtts. Vid den andra snabbare metoden, skakas en bestämd mängd avfall tillsammans med en bestämd volym vätska under 24 timmar. Den vätska som man får efter testerna kan jämföras med ett lakvatten och ska simulera den urlakning som avfallet ger ifrån sig under sin tid på deponin. Denna urlakningsvätska analyseras och de uppmätta halterna av olika ämnen jämförs med en gränsvärdes tabell och avfallet kan hänföras till en avfalls klass.

De två lakningstesterna användes för att karakterisera olika avfallsslag, sönderdelat metall skrot (artikel IV) och slam från gatubrunnar (artikel V). För att få mer kunskap om metoderna och kunna vidareutveckla dem, utökades undersökningarna och analyserna. De urlakade vätskorna analyserades därför enligt Laqua protokollet, dvs med markörer för organiska miljögifter. Dessutom gjordes undersökningar där lakvatten användes som lakningsvätska istället för avjonat vatten som testmetoderna föreskriver. Dessa visade att den mer jonstarka vätskan (lakvattnet) ökade urlakningen av metaller från avfallet. En jämförelse av de två metoderna visade att den snabbare skaktesten oftast gav likvärdiga eller högre halter av de analyserade ämnena i urlakningsvätskan, och därmed kan den i många fall användas i första hand.

För att bedöma ett filtermaterial ur ett livstidsperspektiv, utvecklades en strategi baserad på skaktester (artikel VII). Ett filtermaterial, en mix av torv och aska med kolinnehåll, undersökes före och efter att det använts i en filterbädd för rening av lakvatten. För att vara säker på att filtermaterialet i sig själv inte släppte ifrån sig några föroreningar gjordes en lakningstest. För att se hur effektivt materialet var på att ta bort metaller, PCB och fenolföreningar, gjordes skaktester med vätskor med kända halter av dessa föroreningar. När filtermaterialet är förbrukat och skall bytas ut anses det som ett avfall, och det karakteriserades med lakningstest för att hur det skulle tas om hand. Metodiken med skaktester ger bra information om utlakning från ett material, och skaktester är också bra instrument för att utvärdera ett filtermaterials effektivitet på att ta hand om metaller och opolära organiska ämnen som PCB. Men för att utvärdera effektiviteten av borttagandet av polära organiska ämnen (t ex. fenoler), är inte en korttids skak test något bra instrument. Reduceringen av dessa ämnen sker genom nedbrytning med hjälp av mikroorganismer, och för att undersöka detta behövs tester som varar en så lång tid att en mikrobiologisk miljö hinner etablera sig, t ex. kolonn tester.

Utvärderingen av detta avhandlingsarbete visar på några ytterligare slutsatser och förslag till fortsatt arbete.
Toxicitetstesten med *Artemia* bör kompletteras med tester på t ex. bakterier och växter, eftersom det inte räcker med en test på bara en organism för att bedöma giftigheten av en förorening ett naturligt ekosystem. Vidare behövs en biologisk test för att påvisa kroniska effekter, så som skador på fortplantning eller tumörsjukdomar. Dessa skador kan uppkomma genom att organismer påverkas under en lång tid av de låga, men därmed inte ofarliga halter av organiska miljögifter som ofta förekommer i lakvatten.

Den presenterade metodiken kan användas för att utvärdera miljöfarliga ämnen från olika förorenade områden. Avhandlingen har visat att LAQUA protokollets sammansättning och dess analyser är ett bra instrument för att utvärdera reningstekniker för lakvatten och för att ge ytterligare information om organiska ämnen i fast avfall. Bedömning av luft kvalitet och karakterisering av dagvatten är andra exempel där metodiken kan användas.

Avslutningsvis är det förstås lättare att utvärdera farligheten från avfall när det är mindre volymer avfall att utvärdera. Detta kan uppnås genom att konsumera mindre, återanvända produkter, återvinna material, utvinna energi ur avfallet och välja miljövänliga produkter när man köper nytt.

Till min familj

Max
Lina & Joakim
Emil

Abbreviations

BOD	Biochemical Oxygen Demand
CEN	Comité Européen de Normalisation (European Committee for Standardization)
COD	Chemical Oxygen Demand
DAD	Diode Array Detector
DOC	Dissolved Organic Carbon
EC	European Council
EC_{50}	Effect Concentration (50% response)
ECD	Electron Capture Detector
EU	European Union
EPA	Environmental Protection Agency
GC	Gas Chromatography
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectrometry
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
LC	Liquid Chromatography
LLE	Liquid-Liquid Extraction
LOEC	Lowest Observed Effect Concentration
L/S	Liquid to Solid ratio
MS	Mass Spectrometry
MSW	Municipal Solid Waste
PAH	Polycyclic Aromatic Hydrocarbons
PBDE	Polybrominated Diphenyl Ethers
PCB	Polychlorinated Biphenyls
POP	Persistent Organic Pollutant
PVC	Polyvinyl chloride
RSD	Relative Standard Deviation
SEPA	Swedish Environmental Protection Agency
SFE	Supercritical Fluid Extraction
SLM	Supported Liquid Membrane
SPE	Solid-Phase Extraction
SVOC	Semi-Volatile Organic Compounds
SWM	Swedish Waste Management (Avfall Sverige)
TOC	Total Organic Carbon
UN	United Nations¨
UNESCO	United Nations Educational, Scientific and Cultural Organization
USEPA	US Environmental Protection Agency
VOC	Volatile Organic Compounds

Table of contents

1. General introduction

Consumption generates waste! Economic growth gives increased consumption and hence increased quantities of waste, both from producers as industrial waste and consumers as municipal solid waste (MSW) [1, 2]. Waste management has over the last decades become a serious issue all over the world. It was among the environmental issues of major concern in preserving a sustainable environment on Earth as discussed during the United Nations (UN) conference on environment and development held in Rio de Janeiro in 1992, which resulted in the publication of Agenda 21 [3]. Environmental problems associated with increasing volumes of solid waste are also included in discussions about sustainability and climate change due to global warming caused by emissions generated from human activities. The developed countries have agreed, under the UN Framework Convention on Climate Change (the Kyoto protocol), to reduce emissions of greenhouse gases. Waste management policy will play a part in achieving this objective. The greenhouse gases that make the largest contribution to global warming – carbon dioxide (CO_2), methane (CH_4) and nitrous oxide (N_2O) – are all produced during the management and disposal of wastes. About a third of the European emission of methane can be attributed to landfills. Reducing the amount of methane emitted from landfills would have the greatest effect in reducing the overall impact of solid waste management on climate change. Other environmental and health impacts that derive from solid waste management options include effects attributable to air pollutants such as sulphur oxide (SO_2), dioxins and fine particles, and contamination of water bodies [4].

Waste management is an important part of the infrastructure of society and its optimisation of resource usage. Authorities around the world have established legalisation and guidelines for waste management, to help restrain climate changes and other environmental problems [5-9]. For example, in Sweden climate changes due to effects of waste treatment have decreased by 30% from 1994 to 2005, despite the fact that the quantity of waste has increased by almost the same percentage during this period. This is a result of increased recycling and decreased use of fossil fuels and hence lower carbon dioxide emissions [1].

The European strategy for required actions has been founded on a hierarchy of waste management options established in 1975 [10] and further described in 1993 in the fifth European Community environment action programme [7]. According to the European Council (EC) directive 1999/31/EC [11], the deposition of waste in landfills should as far as possible be minimised in order to reduce the environmental impact. In the sixth European Community environment action programme [8], which provides the strategic framework for the commission's environmental work during the years 2002 to 2012, the management of natural resources and waste is pointed out as one area for priority action. The management options place the first choice on waste prevention. Where waste cannot be prevented, the order of preference decreases in order re-use, recycling, recovery of energy, and finally, as the least preferred option, the disposal in landfills of stabilised wastes from which no further value can be recovered [12].

To create a basis for monitoring compliance with their principles, the Community since 2006 requires statistical measurement data to be acquired by the members according to the EC regulation 2150/2002 on waste statistics [13]. Sweden has implemented these EC directives into Swedish law by issuing the Ordinance on the Landfilling of Waste 2001:512 [14], and the first report originates from 2004. In 2004, around 118×10^6 tons of non-hazardous waste (including 4.5×10^6 tons of MSW), and 1.35×10^6 tons of hazardous waste were produced in Sweden. 62.3×10^6 tons of waste was sent to landfills, most of it (58.4×10^6 tons) has been generated in the mining industry. The amount of MSW sent to landfills decreased by 1 million tons between the years 1994 and 2004 [15, 16]. In 2007, the total amount of MSW collected in Sweden was 4.7×10^6 tons, or 514 kg per person, and the quantity of MSW deposited in landfills was 186×10^3 tons, a decrease of 40×10^3 tons compared to 2006 [17].

Despite all the regulations and developments in waste treatment, there will still be a need to deposit solid waste in landfills. In order to get better control over the type of waste deposited, and over emissions such as leachate and gases coming out from the landfill, criteria and procedures for acceptance of waste at landfills were established in EC decision 2003/33/EC [18]. This was implemented in Sweden in 2005 and can be found in the Swedish Environmental Protection Agency (Swedish EPA) regulation NFS 2004:10 [19]. There were 160 operating landfills in Sweden in 2006 and many of them (about 70) will be closed down by December 31st 2008 when stricter EU regulations come into force [20]. Together with the thousands of old waste dumps that now are disused, these landfills may contain leftovers from everything in the society, including many pollutants that are a threat to human health and the environment.

1.1. Objective

The main objective of the work described in this thesis has been to develop a methodology for evaluating hazards from landfill-generated leachate and from solid waste, and to use this methodology to solve authentic problems. To reach this aim, chemical analytical methods have been refined and technology based on biological markers for assessment of toxicity in leachate has been developed.

Methodology to reach the objective

- Development of a dynamic analytical protocol consisting of chemical and biological analytical methods, for measurements of concentrations of both inorganic and organic pollutants, as well as the acute toxicity in landfill leachate.

Paper I describes the development of chemical analytical methods for organic compounds such as polychlorinated biphenlys (PCB) and phenols, and Paper III describes the development of a biological method for assessment of acute toxicity in leachate, using *Artemia salina* as test organism.

- Application of the analytical protocol to landfill leachate.

Paper II describes how the analytical protocol was applied to the evaluation of the efficiency of different treatment methods in a pilot plant for local treatment of leachate from Härlöv landfill, the MSW deposit situated in Kristianstad, Sweden. Paper VI describes how the same protocol was used for evaluation of treatment efficiency in a lab-scale study of different filter materials on leachate from an industrial landfill containing fragmented metallic waste residuals at Halmstad, Sweden.

- Investigation of the performance of leaching tests on solid waste.

Paper IV describes work on two EU-standardised leaching test methods, and contains also an extension of the procedures directed towards other pollutants and using another leaching liquid.

- Application of leaching tests on solid waste of different types and a filter material.

Paper IV and Paper V describe how the leaching tests were used to characterise and evaluate the hazards of fragmented metallic waste, and sludge from stormwater drainage wells, respectively. Paper VII presents a characterization of a geological filter material, based on a mixture of peat and carbon-containing ash, aimed at leachate treatment. Removal efficiency of different pollutants was considered as well as the leakage of different substances from the filter material, before and after use.

2. Landfills

2.1. Regulations

Emissions from landfills, such as leachate and gas, are well known environmental threats [9, 21] and in order to reduce and control them, new regulations about protective barriers have come into force. The goals of landfill directive 1999/31/EC [11] and the Swedish Ordinance on the Landfilling of Waste 2001:512 [14] are to prevent, or reduce as much as possible, negative effects on the environment from landfilling. Stringent technical requirements are introduced for waste and landfills. Targets for protection are in particular surface water, groundwater, soil, air, and ultimately human health. The regulations define different categories of waste: municipal, hazardous, non-hazardous and inert. Definitions of waste management terms are included in Article 2 in Council Directive 1999/31/EC [11]. The regulation applies to all landfills, defined as waste disposal sites for the deposit of waste onto or into land. These are divided into three landfill classes: landfills for hazardous, non-hazardous, and inert waste. These landfills have different types of protective layers, i.e. a bottom barrier with liners and a top cover, depending on the hazards of the deposited waste. Furthermore, there are some specific types of wastes which are not accepted to be deposited, such as biodegradable municipal waste and liquid waste.

At a modern establishment for waste management in Sweden, only a part of the works is meant for disposal [20]. The main parts of the area at the facility are used to sort as much as possible of the waste in order to re-use, recycle or pre-treat the waste. Places for short-term storage of *e.g.* glass, paper, metals, plastics and refrigerators, and areas for composting of garden material are located at the establishment. Runoff water, airborne particles and gases are emissions which have to be controlled at these places [22-24]. In an investigation performed by the Swedish Environmental Research Institute [25], runoff water from waste sorting areas was found to be the most polluted water compared to other polluted waters, such as treated leachate and sewage. After pre-treatment, the residual waste goes to the landfill for final deposition. The new type of landfills mentioned above must be divided into different cells, designed according to the regulations. According to the acceptance criteria described in EU Decision 2003/33/EC [18] and Swedish EPA regulation NFS 2004:10 [19], waste for disposal must be analysed and characterised based on origin and its leachability, so that it can be allocated to the appropriate type of site. These instructions are explained and practical advice on procedures for reception of waste are given in [26].

New landfills have systems for collection of landfill gases, for example methane, which can be used as fuel for vehicles or for production of heat or electricity. Old landfills often lack these collecting systems. Leachate water is formed when water percolates through the waste in a landfill cell. Modern landfills are built to prevent groundwater from reaching the landfill, and to prevent the leachate from reaching the surrounding groundwater by including liners in the bottom and borders [9]. Suitable constructions for Swedish geological conditions are described in [27]. To fulfil the regulations about how much water is allowed to penetrate through the covering [20], the top cover construction should normally be two to three meters thick in different layers. It should be designed with a sloping surface and the surface runoff should be collected via drainage channels.

Since the beginning of the 1990s several regulations according the producers responsibility have come into force. Some examples are: the EC directive 91/157/EEC on batteries, the EC directive 94/62/EC on packaging, and Swedish national regulations (SFS) such as: SFS 1997:185 on producers responsibility, SFS 2005:209 on electronic equipment, SFS 2007:186 on end-of-life vehicles, and SFS 1993:1154 on packaging of glass and paper [28]. The accepted waste management philosophy of today is “the polluter pays principle”. The main goal is that the producers must pay for the environmental pollution caused by their activities. Accordingly, producers must collect and recycle their products when these are discarded as waste. The objective is to reduce the volume of waste, increase recycling, and encourage environmentally sustainable product development.

The long-term goal for the EU is thus to become a recycling society that seeks to avoid waste, and uses waste as a resource. The proposal is to introduce the lifecycle perspective in policymaking and to clarify, simplify and streamline EU waste law [22]. One very important factor which had made proper waste management difficult is unclear definitions. For example, it is necessary to decide when waste becomes a product for further use. Another important, often discussed question is the definition of what “recycling” really is and what is “end-of-use” [29]. A list of relevant legal frameworks that describe the EU strategy for waste management are available in Annex III contained in the Commission Staff Document SEC(2005)1682 [30].

The number of active landfills is decreasing in Europe as a result of the EC directive coming into force in the member states. However, considering only Sweden, there exist around 5000 closed landfills that still generate emissions [31]. The older closed landfills often lack barrier systems, and the emission potential of these landfills seems to overshadow the benefit of a reduced number of remaining EC directive-compliant landfills [32]. According to the regulations, less biodegradable MSW and liquid waste will be deposited at landfills. This will lead to dryer landfills with slower degradation rates, and thus a prolonged risk of emissions has to be taken into account, when the timescale for aftercare is set [32, 33].

2.2. Waste decomposition in landfills

Knowledge about waste decomposition in landfills has been obtained from control and monitoring programs for existing landfills, and from waste cell experiments [34-36]. Decomposition of waste in a landfill with organic material occurs through a combination of physical, chemical, and most important, biological processes that change the waste composition and emissions. A landfill is expected to exhibit different defined phases of existence, which reflect changes with time in the production and composition of leachate and landfill gases. Easily degradable organic wastes produce landfill gas in the very beginning of the landfill cycle (aerobic phase), when oxygen is rapidly consumed and carbon dioxide is produced. Once the oxygen is consumed, the fermentation processes start (acidic phase), and much of the deposited waste is degraded. The accumulation of carboxylic acids during this stage will decrease the pH of the leachate. This acidic leachate is quite aggressive chemically and often results in an increased mobility of metals. The anaerobic acidic phase can last for several years, but when the pH of the waste becomes neutralised, the methanogenic bacteria start to grow (initial methanogenic phase). The maximum production of methane and carbon dioxide is reached in the stable methanogenic phase. The production of methane declines as the access to degradable material in the wastes decreases, and air will start to penetrate the upper part of the landfill. The more aerobic conditions will drive oxidation of humic compounds and dissolution of metal sulphides [37-39].

2.3. Landfill leachate

As water percolates through the waste body, different groups of compounds are transported in the landfill. These compounds are metal ions and other inorganic compounds, as well as organic compounds that originate from the waste itself, from biological degradation, or from chemical reactions in the waste. Thus, the generated leachate reflects the composition of the waste deposited, and the ageing processes of the landfill, but also the local climate and the design of the landfill. A great number of environmental pollutants are present in leachate. High concentrations of salts, metals, and a huge number of different organic compounds have been reported [21, 36, 38, 40-46]. In a study concerning 12 Swedish municipal landfill sites, more than 90 organic and metal organic compounds, and 50 inorganic elements were detected [40]. Table 1 shows leachate characteristics for several landfills including those investigated in this thesis.

The range for the different parameters can vary by up to two orders of magnitude. This can be seen from leachate data from eleven Swedish landfill leachates compiled from Öman *et al.* [45]. The measured average concentrations of metals in the leachate from the industrial landfill were in some cases above the range. The composition of the leachate from the MSW landfill in Kristianstad is within the same range as for other Swedish landfill leachates. The MSW landfill leachate from Lithuania shows higher values for most of the parameters, and some metals, *e.g.* chromium and nickel, are present at concentrations far above the Swedish range.

Table 1. *Chemical and physico-chemical composition of leachates investigated in the studies described in this thesis and a compilation of leachate data from several leachates, described by Öman et al [45]. Data for leachate from the MSW landfill in Kristianstad, Sweden and in Siauliai, Lithuania are from Paper II, and for the industrial landfill in Hamlstad, Sweden from Kängsepp and Mathiasson [47].*

Parameter / Component	Unit	MSW landfill Kristianstad Sweden average 1993-2002	Industrial landfill Halmstad Sweden average 2003-2006	MSW landfill Siauliai Lithuania December 1999	Range of eleven MWS landfills in Sweden 2000 Data compiled from Öman
$pH_{(KCl)}$		7.2	8.1	8.3	6.4-8.5
Conductivity	mS/m	720	470	1 500	490-2700
Suspended solids	mg/l	140	80	54	8.7-210
Chemical Oxygen Demand, COD_{Cr}	mg/l	660	na	1 500	250-1 300
Biochemical Oxygen Demand, BOD_7	mg/l	27	na	13	4-110
Total Organic Carbon, TOC	mg/l	130*	470	na	52-490
Ammonium, NH_4^+-N	mg/l	250	100	630	93-870
Nitrate+nitrite, NO_3^--N+NO_2^--N	mg/l	0.46	4.0	12	0.18-35
Nitrogen, N-Total	mg/l	270	150	670	100-860
Phosphorous, P-Total	mg/l	1.3	na	4.2	0.2-4.0
Chlorine, Cl^-	mg/l	1600	1200**	4600	360-4900
Calcium, Ca	mg/l	370	30	81	21-340
Iron, Fe	mg/l	5.9***	1.5	na	1.6-43
Arsenic, As	µg/l	5.9*	12	<50	1.3-11
Cadmium, Cd	µg/l	0.2*	0.7	<5	0.06-1.4
Chromium, Cr	µg/l	15	8.4	2 100	4.1-45
Copper, Cu	µg/l	20	190	43	7.0-80
Mercury, Hg	µg/l	<0.1	0.78	<0.1	<0.02-0.1
Nickel, Ni	µg/l	16	76	250	12-91
Lead, Pb	µg/l	3.1*	7.9	<50	1.3-15
Zinc, Zn	µg/l	63	56	170	16-340
Phenol, total (phenol index)	µg/l	57	na	57	na

* Average from raw leachate used in eight weeks pilot plant
** Single value March 2003
*** Average spring 2002
na = not analysed

Many of the compounds have been quantified at very low concentrations, often at µg/l, and a large number of compounds can be expected to be present at concentrations below detection limits of standard analytical methods. However, low concentrations of analysed compounds do not always indicate a less hazardous leachate, since large volumes of leachate can contribute to significant quantities of pollutants released to the environment. Many organic compounds are toxic at very low concentrations, and negative effects on the environment are many times caused by synergistic effects. In addition, non polar, stable compounds are often biologically accumulative, and the concentrations in the environment thus increase with time.

During the last decades, many leachates have been characterised as being generally high in concentrations of nutrients, salts and organic compounds, while being rather low in concentrations of heavy metals. However, leachates from landfills with residuals from incineration have been characterised by higher concentrations of heavy metals and lower concentrations of nutrients. In the future the amounts of these kinds of wastes will increase at landfills and accordingly also the composition of the leachate may change [23]. The presence of a large number of new hazardous compounds in these landfill leachates should have a significant impact on future landfill risk assessments, and in future research it will be necessary to pay more attention to metal–organic and organic compounds [40].

Landfill owners need to have permission for their activities and this includes leachate handling. Determinations of the volume and character of the leachate are the basis for *e.g.* choice of treatment procedures and for risk evaluations of possible toxic effects on the actual recipient. However, there is no Swedish legislation that regulates how the leachate characterization should be done, or how to perform the analyses of the threats from leachate towards the environment and towards human health [48]. Furthermore, there are no national limits or guideline values to compare with. Instead, it is the responsibility of the local operator to assess which parameters need to be included in the characterization program. Accordingly, the stipulated limit values decided at district courts will be different, as can be seen in a report from the Swedish EPA [49]. Due to the large number of environmental pollutants in leachate, there is a need of national guideline criteria, including inorganic parameters, as well as organic marker substances and toxicity tests. Since volumes and components in leachates differ greatly from landfill to landfill, outlet criteria for leachate should be written with both general and place-specific considerations in mind [50].

2.4. Treatment methods for leachate

Control of emissions from landfill leachate includes measurements of volume and composition, treatment and disposal procedures. In Sweden, the amount of collected leachate is estimated to be between eight and twelve million m^3 per year [51]. Most biological, physical and chemical treatment processes used for industrial wastewater treatment have been tested and are in use for landfill leachate treatment [9]. Different treatment methods must be selected for each leachate, due to several factors, such as leachate composition, volumes, emission demands, recipient characteristics, handling of residual products such as sludge, costs and energy consumption. Nearly 100 landfills in Sweden transfer the leachate to municipal sewage treatment plants, either directly or after some kind of local pre-treatment [49]. Table 2 describes different types of leachate treatment systems used at different Swedish MSW landfills.

***Table 2**. Different methods for treatment of landfill leachate reported from Swedish MSW landfills 2003 [49].*

Treatment methods	In use in number of landfills
Aerated pond	86
Municipal sewage treatment plant	84
Irrigation of soil-plant system	37
Re-circulation to landfill	29
Infiltration	28
Overflow	18
Filter-bed	14
Chemical precipitation	9
Sequenced batch reactor (SBR)	4
Mechanical treatment	4
Wetlands	*

* Wetlands are not separately described but might be involved in some of the methods listed above.

One possibility for treating leachate is to pipe it to a nearby sewage treatment plant. Unfortunately, there are problems with mixing leachate and sewage. The composition of leachate water is quite different compared to sewage. The municipal sewage treatment facilities are normally not adjusted for the composition of pollutants that occurs in leachate, which might result in inhibition of respiration or nitrification processes in the treatment plant [52, 53]. Furthermore, the sludge emanating from the treatment process is rich in nutrient, and in a sustainable society this sludge should be used as a fertiliser for agriculture. However, in recommendations from The Federation of Swedish Farmers (LRF), nutritive substances that are directed towards agriculture are not allowed to be contaminated with metals or organic pollutants [54]. This means that if leachate is treated together with the municipal wastewater, the sludge from this treatment plant can not utilized in agriculture. Accordingly, local authorities in Sweden strive to find other solutions for landfill leachate treatment. About 140 landfill owners in Sweden have some kind of local treatment of the leachate, either as a separate system, or as pre-treatment before transfer to a municipal sewage treatment plant [49].

Different systems for treatment of leachate are in use. Some of the most commonly used systems in Sweden are briefly described below. More details about the efficiency and applicability of different methods can be found in Refs. [25, 48, 49, 55-57].

Many treatment systems use both physico-chemical processes and biological degradation. Storage, catching and adjustment in ponds are often used as pre-treatment steps. The ponds can be placed in series and one of these ponds is then generally aerated. The flow through the system is low to enhance sedimentation of particles. Natural treatment based on a soil-plant system has been in use in Sweden since the mid-1980s. More than 40 landfills had such systems in 2006 and additional such systems are planned. These systems, with irrigation or overflow of wastewater over plants such as willow or energy grass, are aimed for reduction of the leachate volume and for consumption of nutrients, such as nitrogen, through conversion to plant biomass [46]. In constructed wetlands, the ammonia reduction is usually high, since such systems generally use both aerated and non-aerated areas to promote nitrification as well as de-nitrification processes. In filter-bed systems, water is spread over a bed of geological material, such as sand, peat or bark [47], and the water percolates through the filter-material. Processes like separation, biological degradation and sorption occur, which results in reduction of particles, nitrogen, organic compounds and metals in the leachate. Other biological treatment systems are bioremediation systems with active sludge, like sequenced batch reactor (SBR) or biological filters. These systems have a biofilm of microorganisms attached to a carrier, which gives good reduction of easily degradable organic materials and nitrogen-containing compounds. Other techniques used for wastewater treatment, but not common in Sweden for leachate treatment, are chemical precipitation, active carbon adsorption, reverse osmosis and ozonation. Effective removal of organic compounds from leachate has been found in studies using these methods alone or combined with other methods [58-61].

The choice of treatment technique or combination of techniques is dependent on leachate characteristics and landfill location. In the pilot plant at Härlöv landfill at Kristianstad, described in Papers I – III, several procedures were tested. These were pre-treatment in ponds (two aerated and one for sedimentation), followed by one of four different treatment procedures: bioremediation, ozonation, chemical oxidation by Fenton's reagent, or the use of filter-beds (see Paper II). Leachate treatment in filter-beds with different filter materials such as peat, carbon-containing ash, wood pellets and polyurethane were studied in the lab-scale test described in Paper VI.

3. Leaching tests

There are different ways to measure pollutants when evaluating the environmental threat from a solid material. The question is which is of greater importance: the total amount of a pollutant in a material, or the biological available amount of the pollutant, or the amount of pollutant that can leak out to the water [62]. To estimate the environmental threat of solid materials, leaching tests are central tools for material characterization. Leaching tests are considered to yield more relevant information than, for instance, a determination of component composition [63]. In the simplest form of leaching test the test material is brought into contact with a liquid (leachant) under controlled conditions and different parameters are analysed in the resultant liquor (eluate) [64]. Further questions then concern how much of the pollutants leak out during the first rainfall, how much will come out within a few years, and what amount will leak out during the whole lifetime of the material (i.e. until it is totally degraded). This will determine the hazard of interest for the particular situation. The liquid-to-solid ratio (L/S-ratio), i.e. the ratio between the amount of liquid (L in litres) which had been in contact with the material, and the mass of the solid material (S in kg of dry matter), is a key factor in a leaching test for these evaluations. With knowledge about precipitation, the mass of material, its hydraulic conductivity and the thickness of the layer, the L/S-ratio can be transformed into a timescale [62].

Bench-scale leaching tests were first used during the early 1980s for assessing the environmental impact of solid waste disposal to landfills. Numerous different leaching procedures have since then been developed [65]. Leaching is a complex phenomenon determined by many factors, which may influence the release of specific constituents from a waste over extended time intervals. These factors include major element chemistry, pH, complexation, redox potential, liquid-to-solid ratio (L/S), contact time and biological activity. Different leaching tests focus on different factors, and the purpose of the test should determine which test that should be chosen [66]. A hierarchy of tests with distinction between tests for waste characterization, compliance and on-site verification, has been developed within the framework for standardization by CEN, the European committee for standardization:

- Characterization tests are primarily used for basic characterization of the release behaviour of the material.
- Compliance tests have the purpose to check whether a material has the same leaching behaviour as a reference material (tested with a characterization test) and/or complies with regulations.
- On-site verification/quality control tests have the purpose to determine in a short time if a material matches with earlier determined or expected behaviour in its practical application. In general, in this test only administrative checks and visual control will be done [67].

The increased use of widely different leaching test methods in different areas has led to a harmonisation project, which started in the mid nineties by the CEN commission.

The summary Paper from the initial workshop presents a list of different methods used by the participants [68]. A network was established [69] and test methods have been validated and approved by the CEN [70].

3.1. Types of tests

Many studies have been presented in when different leaching tests have been compared and evaluated [62-64, 70-73]. Table 3 shows a compilation of the most frequently used leaching test methods together with a description of the purpose and usage of the tests.

Table 3. *Leaching test methods, examples of national standards and description of purpose and usage of the tests. The summary is compiled from [62, 72].*

Leaching test	Describtion	Ex. Methods
Column test	Characterization of waste - Up-flow percolation test	SIS-CEN/TS 14405 1)
Batch test	Charaterization of waste - Compiance test for leaching of granular waste materials and sludges. One- or two-stage shaking tests	SS-EN 12457 1-4 1) TCLP Method 1311 2)
Diffusion / tank test	Determination of leaching from building and monolitic waste materials during a longer time	NEN 7345 3)
Availability test	Method for creating a leaching optimum of a compound over a long time. The results are regarded as the maximum leachable amount	NT ENVIR 003 4) NT ENVIR 006 4)
pH-static test	Basic characterization test. Gives a good view of the influence of change in pH obtained in the leachate	SIS-CEN/TS 14429 1) SIS-CEN/TS 14497 1)
Lysimeter test	Pilot scale experiments, naturally produced leachate is collected and analysed. Used to verify results from laboratory tests and investigate divergence in the field situation.	

1) Swedish and European standard

2) US standard

3) Dutch standard

4) Nordic recommendation

The EU technical standard percolation test (SIS-CEN/TS 14405:2004) is performed in columns and demineralised water of natural pH is used for the leaching. Concentrations are usually measured in 7 different fractions up to cumulative L/S 10. The choice of L/S 10 is often representative for a long-term situation in practice. The test is designed in such a way that local chemical and physical equilibrium is attained [67]. This column test should be used for basic characterization of a waste.

The EU-standard batch test (SS-EN 12457 1-4) consists of four parts, each describing a compliance test, but with differences in L/S ratio and grain size. The leaching test is performed by adding a mass of material to a desired volume of leachant (depending on the chosen L/S ratio). The leaching is carried out in bottles mounted on a shaking table agitated for 24 hours. This EU-standard is based on the assumption that equilibrium or near-equilibrium is achieved between the liquid and the solid phases during the test. The solid residue is separated by filtration [74]. Batch tests are carried out as single batch leaching tests at fixed L/S ratios of 2 or 10, or as two-stage batch tests at L/S 2 and 8 to give a cumulative L/S ratio of 10. This batch test should be used in the compliance test of a waste.

The pH-static tests (*e.g.* SIS-CEN/TS 14429:2005, SIS-CEN/TS 14497:2007) are carried out at L/S 10 and various amounts of acid and base are added to obtain the final pH. After equilibrium periods of 48 hours, concentrations of elements are measured in the eluates. The methods give a good view of the influence of a possible change of pH obtained in a leachate. The methods can be a tool for indication of long-term leaching from waste, where changes in pH may be considerable. The test also gives an indication of the buffer capacity of the waste [67]. The test is designed to represent chemical and physical equilibrium conditions, and may be used for basic characterization of waste.

The availability tests (*e.g.* NT ENVIR 003, NT ENVIR 006) estimates the fraction of a component available for leaching in a geological time frame, i.e. the possible amount that can be leached out when particle size, alkalinity, differences in concentration or time do not limit the leachability. The tests give an estimate of the long-term leachability and the results may be used as the upper limit for possible leaching. The availability test is performed with granular material and with a leachant at pH 4 and pH 7. The test is primary used for basic characterisation, but it can also be used for compliance testing [62].

The landfill directive (1999/31/EC) requires separate disposal of waste to landfills for inert, non-hazardous and hazardous wastes and gives the waste acceptance criteria (WAC) for these different classes [11]. The EC decision 2003/33/EC [18] describes a three-level procedure for the acceptance of waste to landfills, and provides specified use of testing methods. For leaching tests the up-flow percolation test, SIS-CEN/TS 14405:2004 [75], and the batch test, SS-EN 12457 [74] are provided. The criteria provided apply to eluates generated at L/S 2 and/or L/S 10 from the batch leaching test, and/or initial C_0 eluate concentrations, L/S 2 and/or L/S 10 from the percolation test. The criteria include limit values on the leachability for a number of predominantly inorganic parameters [76].

The column leaching percolation test is a so-called characterization test and can give information on leachability over short and long time periods. The batch shake test is a compliance test and has the purpose to check whether a waste material complies with regulations. The experimental part of the percolation test takes a longer time, requires more advanced equipment and more manual work than the two-stage batch test. According to the landfill directive, both tests can be used for assessment of leachability for medium and long times, and both tests can be used for compliance testing of waste after a basic characterisation has been done. In the work described in Papers IV and V, these two EU standardized leaching methods were studied and the concentrations of parameters in the eluates were compared for the two fractions L/S 2 and L/S 10.

3.2. Leaching of organic compounds

The understanding of leaching phenomena and the access to suitable methods for determination of inorganic components from soil and waste have increased during the past decades. However, many of the contaminants of interest are organic compounds, and practical experiments with testing the leaching of organic compounds are limited. In a Nordtest project, an evaluation was made of existing methods on leaching of organic compounds from contaminated soil and waste materials [77]. During this project some organic compounds, such as benzene, toluene, ethyl benzene and m-xylene (BTEX), polynuclear aromatic hydrocarbons (PAHs), PCBs, chlorinated phenols, and chlorinated solvents, were identified. These should preferably be included in a leaching test. Some general and specific conclusions, and recommendations about e.g. equipment, are also described in this report [77]. The European regulation [18] and the Swedish legislation [19] include waste acceptance criteria for the acceptance of waste at each landfill class (inert, non-hazardous and hazardous waste). These criteria include waste leaching limit values for organic sum parameters, *e.g.* total organic carbon (TOC), dissolved organic carbon (DOC), BTEX, PCBs (sum of 7 congeners), mineral oil (C10 to C40), PAHs and phenol index for acceptance to landfills for inert waste. At present, criteria for landfills for non-hazardous and hazardous waste include only leaching limit values for organic sum parameters.

3.3. Leachant

The leaching of contaminants from waste is controlled by several parameters and external factors. One of these is the nature of the leachant. For most waste types, the composition of the eluate at L/S values below 10 l/kg is controlled predominantly by the composition of the waste [75]. According to the Technical Standard it makes little difference whether the leachant consists of de-mineralised water or rain-water (real or artificial). For the sake of simplicity the percolation test recommends demineralised water with a conductivity of a maximum of 0.1 mS/m as leachant. For specific purposes it is possible to use other leachants. The EU-standardized batch test method [74] recommends water with pH value between 5 and 7.5 and conductivity below 0.5

mS/m. The choice can be distilled water, demineralised water, de-ionised water or water of equivalent purity.

The pH value of leachant is of great importance for the leachability of a compound [62]. For investigation of extraction of metals from waste material, acidic leachants are recommended, as for example in the pH static tests and in the US standard toxicity characterization leaching procedure (TCLP) [78]. The release of contaminants from waste is highly correlated with the final pH in the eluate. Studies with acidic solutions as leaching medium, to simulate acid rain water, have been presented [63, 73, 79, 80]. From these studies can be seen that the final eluate appeared to be a function of the interaction between the waste pH and the amount of acid available in the leachant to react with the waste. Increasing L/S ratios shifted the eluate pH towards the pH of the leachant. When metal content is high in the waste material, an acidic leachant can affect the concentration of metal leached.

Under natural conditions, water percolating through the waste body gives leachate water with high ionic strength. This type of water is in fact what the investigated material comes in contact with after its disposal at the landfill. However, this type of leachant is rarely investigated in leaching tests. There are studies using leachants other than demineralised water or acidified liquids, *e.g.* synthetic leachate [79] and weak ionic solutions [81]. Natural leachate, with generally high concentrations of chloride and ammonium ions, may impact the leaching of heavy metal ions from the waste material. Heavy metal ions may be released from solid waste because of strong interaction with chloride and ammonium ions. In the investigation of the leachability of fragmented metallic waste described in Paper IV, the performance of the two test methods was extended by also using natural landfill leachate as leachant.

4. Analytical protocol

Environmental science is based on analytical know-how including monitoring of environmental pollutants. Some specialists suggest that there exists a separate field of analytical chemistry named ecoanalytics [82]. Both analysis of individual samples and more or less continuous environmental monitoring are powerful tools that can give information required for evaluation of the level of environmental contamination, and the changes taking place in the environment, necessary for making correct decisions and taking effective action. New methodology and equipment with better reproducibility, repeatability, and detection limits have increased the possibility to identify pollutants and to measure their concentrations in different parts of the environment, such as air, water or soil [83]. The strategies for the analytical work vary depending on the purpose and can be divided into different categories [38, 82]. When known emissions and known treatment procedures are monitored, as in sewage treatment plants or factory effluents, quality and emission control can be performed using existing standard methods, and the results can be related to historical data and regulations. Established methods, with grab sampling in a limited geographical region, are generally used for identification of sources of pollution and determination of emissions. Environmental screening, in order to evaluate the occurrence of new or known suspected pollutants, may cover an expanded national or even world-wide scale. To explain mass transfer of pollutants in order to estimate the environmental impact, i.e. examination of the effect of pollutants on climate change, passive sampling technology is a growing area [84, 85].

4.1. Environmental pollutants

A great number of environmental pollutants arise during waste handling, as previously mentioned. High concentrations of salts, metals and a huge number of different organic compounds have been reported in leachate. A good description of well-known toxic compounds concerning their usage and threat to environment and human health has been made by Sterner [86] and Baird & Cann [87]. Pollutants of special interest in waste management are briefly described below.

4.1.1. Metals

All metals are potentially very toxic. Many poisoning and environmental catastrophes are related to bad handling or control of metals The greatest environmental hazards are considered to come from arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb), mercury (Hg) and nickel (Ni). This is related to the extensive use, toxicity, and widespread distribution of these metals. Although mercury vapour is highly toxic, the other heavy metals are not particularly toxic as condensed free elements, but they are all dangerous in their cationic forms and are also highly toxic when bonded to short chains of carbon atoms. Transport of heavy metals through air occurs either as gases or as species trapped in or adsorbed on particles. But the ultimate sink of heavy metals is soil and water sediments. Thus, humans are mainly exposed to heavy metals through contaminated food. One example was the mercury poisoning of fish in Minamata, Japan in the 1950s. Another was the itai-itai disease, caused by cadmium-contaminated rice. These events were related to metal-contaminated water bodies, due to release to a recipient of contaminated wastewater from industry. Plants got contaminated through absorption from irrigation with contaminated water and by uptake from contaminated soil [87].

Distribution of metals to the environment will most probably decrease with increased sorting of waste. Recycling of metals is both economically and environmentally favourable and less metal will certainly be deposited in the future. Even the regulations on collection of electronic equipment will probably help to reduce metals in the environment. However, large volumes of products made of metals have been deposited over many years, and metal ions will continue to leak from them for many decades. Thus, metals still have to be removed from the leachate before it reaches a recipient.

Modern analyses of metals are performed using inductively coupled plasma, with either atomic emission spectrometry (ICP-AES) or mass spectrometry (ICP-MS). ICP-MS gives very precise and accurate measures even at very low concentrations (at sub ppb level) and is widely used, although the equipment is expensive [88].

4.1.2. Organic compounds

Chemists have learned to synthesize a vast number of chemicals in the last decades, many of them non-existant in nature. Many of these have a strong positive impact on human well being, but many have also been shown to be toxic, causing severe environmental pollution. Synthetic chemicals can be divided into pesticides and non-pesticides. The last group is most important from a waste management perspective, since such chemicals are used in fabrication of products and may thus be emitted after their usage.

Pesticides are substances that kill or otherwise control an unwanted organism. These substances are divided into further categories depending on their target organism, and the three most used are insecticides, herbicides and fungicides. The organic pesticides were initially thought to be biodegradable, but this has in many cases been found not to be the case, and the main portion of organic compounds listed by the United Nations Environmental Program as Persistent Organic Pollutants (POPs) are organic pesticides [89]. Non-pesticides are a group of organic compounds that end up in the environment due to pesticide manufacture or other anthropogenic processes.

The environmental pollution by POPs like dioxins, furans, PCBs, and other organochlorines belonging to the class of POPs, are known to be high, and the discussion about the effects on human health will continue. These persistent organic pollutants are hydrophobic and have a tendency to dissolve in fatty matter and therefore bioaccumulate in the food chain. The highest concentrations can be found in top consumers like birds of prey, seals and humans. It is generally believed that most of them are not acutely toxic to humans, but scientists are more worried about the long-term effects of exposure. Humans are nearly always exposed through water or food, especially limnic fish. Researchers also stress the fact that there is a strong bioaccumulation and high toxicity related to these substances in almost all environments, even far away from centres of human activity. They consider that their harmful effects, such as cancer and birth deformities in wildlife, must be a warning signal for humans. On the other hand, there are researchers who claim that the dangers from these chemicals have been overrated. They point at the very low concentrations in the environment and the enormous economic costs associated with remediation processes [87].

Polychlorinated biphenyls, PCBs, stands for a group (consisting of 209 congeners) of industrial organochlorine chemicals. Large quantities of PCBs have been produced in the world since the late 1950s. Sweden totally banned all new usage of PCBs in 1978. PCBs are practically insoluble in water but are soluble in hydrophobic media like animal fat. They were very attractive commercially in electrical insulating because they are difficult to burn and inexpensive to produce. PCBs can also be found in plastic products and demolition materials, because they have been used as softening agents in polymers, paints and joint material in building constructions. As a result of their stability and extensive use, in combination with careless disposal practice, PCBs have become major environmental pollutants in many areas of the world. There are measurable background levels even in polar regions and at the bottoms of oceans [87].

Polybrominated diphenyl ethers, PBDEs, are a group organic compounds (consisting of 209 congeners) commonly used as fire retardants. They are used in products such as polyurethane foam, textiles, ready-made plastic products and certain electronic equipment, for prevention of fire. The least toxic PBDE is a fully brominated congener, almost the sole ingredient in the commercial product named Deca. Some scientists suspect that deca-PBDE may also degrade in the environment by loss of some bromine, and thereby dramatically increase the toxicity of this commercial product. The commercial products Octa and Penta, which are banned in the European Union, include congeners with fewer bromine atoms [87].

Dioxins are highly toxic by-products which are formed in waste incinerations plants during combustion of products containing organochlorine compounds, *e.g.* PVC plastics. Nowadays there is a good knowledge about how dioxin is formed in different combustion processes. These can be regulated and the problem with dioxin in the environment will probably decrease in the future. *Furans* are produced by strong heating of PCBs in the presence of oxygen. The threat to human health from furans in the environment may exceed that from dioxins, since incineration produces a greater mass of furans than of dioxins.

Polynuclear Aromatic Hydrocarbons, PAHs, is a group of hydrocarbon molecules which consists of two or more six-membered benzene-like (aromatic) rings. The simplest example is naphthalene, the only one manufactured commercially. PAHs are formed when carbon-containing materials, like fossil fuels, garbage and wood, are incompletely burned. A substantial quantity is generated in the production of the wood preservative creosote.

Phenolic compounds are aromatic alcohols with a hydroxyl group (-OH) coupled directly to the benzene ring. Phenols are, as other alcohols, in general relatively water soluble. Phenolic compounds in the environment have both natural and anthropogenic origins. They are produced by plants, but they are also used as industrial bulk chemicals and in preparation of solvents and disinfection agents. Consumer products which can contain phenols are *e.g.* pharmaceutical preparations, paint removers and synthetic resins [90]. Cholorinated phenols (consisting of 19 compounds) have been important in production of biocides and wood preservatives, and can for example be found in pressure-creosoted timber.

The analytical effort in detection and monitoring of organic compounds is complicated, time consuming and costly. Usually a methodology based on different kinds of chromatography, gas chromatography (GC) or liquid chromatography (LC), is used. A large variety of detectors exist for both LC and GC, utilising different chemical or physical properties of the analyte in order to produce a measurable and quantitative signal. In trace analysis using GC with an electron capture detector (ECD) or mass spectrometric (MS) detector are most common. In LC, the most common detectors are ultraviolet (UV) detectors where the absorbance is measured for a specific wavelength or for a whole wavelength range using diode array detector (DAD), and MS detectors [88].

Analysing organic compounds generally requires some kind of pre-treatment of the sample, usually referred to as sample preparation. This includes steps like filtration, pH adjustment, distillation, analyte trapping, acidic degradation, evaporation, and a variety of extraction procedures. Sometimes one step is sufficient, but often a combination of several preparation steps is required. Usually the concentrations of the interesting organic compounds are very low in environmental samples, and are often masked by other compounds. The purpose of sample preparation is in most cases to both remove interferences and to pre-concentrate the analytes into a phase suitable for the selected final analysis [38]. Liquid-liquid extraction (LLE) has historically been the most used extraction method for organic compounds, but there are drawbacks with LLE. The major disadvantage is the need for large amounts of solvent. A more recent technology is the solid phase extraction (SPE) method, where a partitioning of the analytes occurs between the aqueous sample and a solid phase sorbent, often a C_{18} bound silica phase. When rinsing the analyte from the solid phase, large amount of solvents are used. An alternative to organic solvent elution is to use supercritical carbon dioxide delivered by an automated apparatus [91].

4.1.3. Inorganic compounds

In an ecosystem in balance, nutrients – especially nitrogen and phosphorus – are required for production of biological material. However, if large amounts of nutrients are discharged into the environment, eutrophication may occur and severe environmental problems will follow. The main sources of these nutrients are runoff water from fertilised agriculture land and from incompletely treated wastewater from municipal and industrial treatment plants [92].

Nitrogen compounds differ in the extent of oxidation of the nitrogen atom and are in different ways important for aquatic living organisms. The most reduced form of nitrogen occurs in ammonia, NH_3, and its conjugate acid, the ammonium ion, NH_4^+, and both forms are found in water. The proportion between them depends on pH, where higher pH shifts the equilibrium towards ammonia. It is mainly the non-ionic form, ammonia, which causes toxic effects on aquatic life. By the aerobic microorganism-catalysed process of nitrification, ammonia and ammonium ion are oxidised to nitrate, NO_3^-. The most important intermediates are the nitrite ion, NO_2^- and molecular nitrogen, N_2. In the anaerobic de-nitrification process, nitrate and nitrite are reduced to nitrogen gas, which will evaporate to the air. In construction of a treatment plant for wastewater, the focus for reduction of nitrogen in water is transformation of nitrogen compounds from NH_3 / NH_4^+ to NO_3^- and further to N_2.

Phosphorus mainly enters the lakes as dissolved inorganic compounds. The phosphate ions, PO_4^{3-}, are often absorbed to small colloidal particles and a major loss of phosphate is by precipitation.

There are several standardised analytical methods for detection and quantification of ionic compounds. Liquid chromatography based on separation of the ions (IC) followed by a conductivity detector, is a fast and frequently used methodology. Spectrophotometry is another commonly used methodology that uses absorption of light to measure chemical concentrations. Many ready-to-use kits, with different colour reagents for fast and easy routine analyses of nutrients are available on the market.

4.2. Biological testing

Control of wastewater effluents has traditionally been regulated by some of the above described chemical methods, but these give no information about biological effects. Therefore, during the last decades, there has been an increased interest in correlating chemical analysis with toxicity data in order to increase our ability to assess the environmental impact of different wastewater effluents [40, 93-98]. There is a strong need for rapid, simple, sensitive, and at the same time cost-effective analytical strategies. Consequently a wide range of biological toxicity tests (biotests) have been developed in order to predict the effects of pollutants on aquatic biota. Biotests can be based on microorganisms, invertebrates, plants, fish or other bioindicators. A summary of different methods and their usage, advantages and disadvantages, can be found in Farré and Barceló [99]. The US EPA has issued guidelines to assess whole effluent toxicity (WET) which include measurements of toxicity, both acute and short term chronic effects, of wastewater effluents that are required to be monitored due to their potential environmental impact [100].

The general approach for measuring toxicity in water samples is to incubate a number of organisms in a series of successively diluted samples. After a defined incubation period the number of organisms suffering a defined effect (*e.g.* death, growth inhibition, immobility, etc.) are recorded. The result is generally reported as EC_{50} (effect concentration) or LC_{50} (lethal concentration), the concentration that affects or kills half of the tested population [101]. The results can also be given either as lowest-observed-effect-concentration (LOEC), the lowest concentration of the toxicant for which a statistically significant effect is observed, or as no-observed-effect-concentration (NOEC), the highest toxicant concentration for which no statistically significant effect is observed.

A biotest should yield some sort of ecological data, but this is seldom obtained with one single organism, since very little can be said about the effects on a whole system from data from just one organism. Thus, the optimal test would be a battery of biotests, which utilize organisms representing different trophic levels in the ecosystem [43, 102, 103]. Depending on the organism and on the purpose of the test, different responses will be measured. For acute toxicity, a defined effect (*e.g.* death, growth inhibition or immobility) is measured after a limited time, from 5 to 30 minutes for bacteria up to 24 to 96 hours for crustaceans. Chronic or reproductive toxicity tests study the effects in a life-cycle perspective of the organism or the genetic changes in the cells. A battery of tests can also be composed of acute, sub-chronic and genotoxicity tests, in order to even get information about the bioaccumulation and persistence of organic compounds in the wastewater [44].

Another interesting approach is to use bacteria-based test systems in a micro-plate. Microorganisms are often tolerant to a much wider range of environmental parameters than animals, and there are far fewer ethical and regulation problems regarding usage of microorganisms in toxicity tests. High numbers of individual cells can be used, and coupled with short generation times this shortens the required testing time. Single strain bacteria-based toxicity assays have been presented by Bitton *et al.*[104]

(commercial name Met-PLATE^{TM}) and Botsford *et al.*[105]. The Met-PLATE^{TM} was investigated in a study for the specific determination of metal toxicity and heavy metal binding capacity of MSW landfill leachate in Florida [96]. However, even more interesting is the approach with different microorganisms in the same micro-plate, the multi-species microbial assay for risk assessment (MARA), presented by Gabrielson *et al.* [106]. The MARA method is based on measurements of microbiological growth inhibition due to exposure to different toxicants, such as phenol and pentachlorophenol, and a diverse pattern of toxicity values – "a toxic fingerprint" – from each tested chemical is generated. The use of MARA in complex wastewaters is not yet investigated.

Chronic toxicity can be measured by monitoring changes in *e.g.* growth, reproduction, hatchability and genetic changes of organisms exposed to the polluted water for a longer period of time, up to a generation cycle of the organism. The concentrations of the pollutants should be in the range of what will be expected at the outflow to the recipient [45]. One disadvantage with chronic tests is that they generally take a long time, require considerable manual work, and thus will be expensive. During the last decade, environmental immunoassays have been developed to detect selected pollutants in water. Several commercial kits are available and offer rapid screening at comparatively low costs. Methods such as enzyme-linked immunosorbent assays (ELISAs) can be very specific to a certain compound or a group of compounds, *e.g.* PCBs [107]. Fishes and mussels are often used as biological indicators for assessing environmental pollution in natural waters. In an environmental monitoring program for surface water, physiological and bio-chemical investigations of *e.g.* tissue, blood or body fluids from this type of organism are included. In these investigations, the organisms are collected from the investigated water body. However, these test methods are also suitable to perform in laboratory scale under controlled conditions. Sedentary and filter-feeding mussels take up and concentrate contaminants to levels above those in the surrounding water. Examination of their tissues gives information on the bioavailable fractions, which may cause harmful effects [108]. Fish are generally considered to be the most feasible organisms for monitoring pollutants in aquatic systems. They can be found virtually everywhere in the aquatic environment and they play a mayor ecological role in the aquatic food-chains [109]. In order to assess exposure to, or effects of, environmental pollutants on aquatic systems, a set of different fish biomarkers are available. For example, measuring the induction of vitellogenin in male fish provides the basis for a biomarker assay for studies of endocrine disruptors [110].

The interest for biosensors in environmental monitoring is growing. Biosensors are analytical devices that combine a biological sensing element – such as enzymes, antibodies, cell receptors, tissues etc. – with a transducer, which converts the biological signal into a measurable physical signal. The increasing interest for development of biosensors for assessing toxicity is due to the possibilities of mass production, ease of use, fast response, and adaptability to on-line monitoring [99].

To assess the toxicity from leachate, several different biotests using organisms from different trophic levels, *e.g*. bacteria, algae, crustaceans, plants and fishes, have been used [43-45, 111-113]. Commonly used methods are Microtox® (based on the luminescent bacteria *Vibrio fischeri*) [114], the growth inhibition test (based on the green algae *Raphidocelis subcapitata*) [115, 116], the acute- or sub-chronic test with the crustaceans *Ceriodaphnia dubia* [117] or *Daphnia magna* [118] or the rotifer *Brachionus calyciflorus* [119], and the genotoxic umu-test (based on the bacteria *Salmonella typhimurium*) [120]. When assessing toxicity in complex wastewater such as leachate, there is a risk that common water quality parameters such as pH, alkalinity, salinity etc. can mask the toxic effects from organic compounds of more environmental concern [43]. Since the salinity in landfill leachate is generally quite high, it may influence the toxicity for freshwater organisms such as *Raphidocelis subcapitata*, *Ceriodaphnia dubia* and *Daphnia magna*. Therefore, a toxicity test method for landfill leachate using the brackish water crustacean *Artemia salina* was developed and presented in Paper III.

5. Methodology and applications

The initial work was carried out within a project called LAQUA, financed by SweBaltcop, a European Commission programme for Baltic region cooperation. The project had the objective to develop ecologically and financially sustainable methods for local treatment of leachate from waste deposits. Accordingly, the first and main objective of this thesis was to set out a methodology to evaluate pollution problems related to landfill-generated leachate (Papers I, III). Later on the objective was extended towards development of a methodology based on leaching tests, which would make possible a proper classification of solid waste. The developed methods have then been used in three different types of studies, namely, investigation of different treatment steps in a pilot plant and in a column study (Papers II, VI); evaluation of leaching tests (Papers IV, V); and investigation of new and used filter materials for leachate treatment (Paper VII).

5.1. The LAQUA protocol

With the increasing amount of pollutants generated in our society, different wastewater streams, such as leachate, stormwater and sewage, contain a large number of chemical compounds [25]. However, due to high-costs and time-consuming analyses, it is not possible to analyse all these compounds. On the other hand, general organic sum-parameters, such as chemical oxygen demand (COD), biochemical oxygen demand (BOD) and total organic carbon (TOC), hardly provide enough information about the organic constituents in the monitored waters. To improve this situation the LAQUA protocol was developed as presented in Paper I. This protocol is based on determination of a limited number of marker substances and includes a biomarker, which gives an estimate of total acute toxicity of a sample. The LAQUA protocol is especially valuable when considering changes in wastewater composition after a treatment step. In this case relative quantitative values, obtained with good accuracy, give sufficient information. The LAQUA protocol should be seen as a dynamic procedure. The ingoing analytical parameters may be changed, depending on what kind of wastewater is to be studied. The dynamic LAQUA protocol is shown schematically in Figure 1.

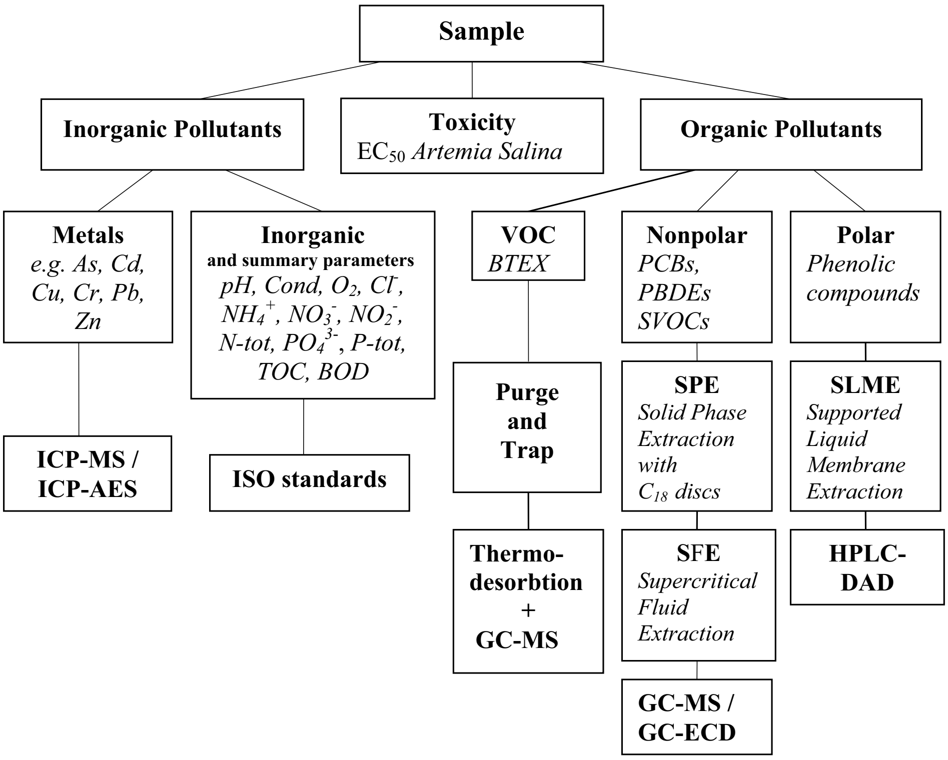


Figure 1*. The LAQUA protocol: an analytical strategy for characterisation of leachate and evaluation of treatment procedures.*

The protocol is divided into three main sub-parts which cover measurements of inorganic and water-quality parameters, organic compounds, and toxicity, respectively. The protocol also describes sample preparation and type of analytical methodology for different substances. For non-polar compounds the SPE step can be followed by an elution step using organic solvent, which however may give lower selectivity. The sampling process is very important in obtaining representative samples. A time-integrated sampling method was used in the pilot plant study (presented in Papers I-III). Integrated sampling is necessary in order to compensate for fluctuations of the inflow to a treatment plant, due to factors like precipitation and construction of the drainage system.

5.1.1. Inorganic and water-quality parameters

It is necessary to evaluate general inorganic and water quality parameters to be able to get good basic data for any action program. General organic sum parameters such as BOD and TOC are included among the water quality parameters. These methods mainly provide information on the total organic content but give very little information on the identity of actual organic constituents in the water. However, BOD and TOC should be included in general characterization of the water for historical reasons, since they provide data that can be compared to old data. TOC values also provide a possibility for an evaluation of the overall level of pollution in the water, and are useful for generally evaluating the results of different treatment techniques. The measurement of COD is left out from the protocol because of the required use of mercury for removal of interferences from chloride, present at high concentrations in leachate. The non-specific analytical response from COD determination also justifies the exclusion [82].

Measurement of all nitrogen parameters is done, since this is important in understanding the effects of the treatment procedure. As has been shown in Paper II, increasing efficiency towards removal of organic pollutants is obtained in biological treatment procedures, as the extent of transfer of ammonia to nitrate and nitrogen gas increases.

Metals always need to be monitored, due to their occurrence and known adverse environmental impact. The selection of which metals to monitor should be based on metals of expected interest, which might be obtained by a screening of the water. The described studies in this thesis have all utilised ICP-MS and ICP-AES for analyses of metals in water and soil.

5.1.2. Organic compounds

Leachate contains a vast number of organic compounds, including PAHs, pesticides, phtalates, PCBs, PBDEs, a variety of phenolic compounds and other priority pollutants [40, 45]. It is not convenient, or necessary, to measure all organic compounds which could ultimately be detected by HPLC or GC analyses to be able to evaluate the efficiency of a certain treatment step. These analyses would take a very long time to perform and would be very costly. Instead, a strategy is needed in which groups of compounds or even single compounds can be used as markers for the given present situation in the leachate. During the development of the LAQUA protocol (Paper I) classical organic parameters such as amounts of PCBs and phenolic compounds were selected for monitoring. Furthermore, supported liquid membrane (SLM) extraction for sample preparation was introduced, to reduce the amount of expensive and high quality solvents needed. By combining SLM with high performance liquid chromatography (HPLC), the whole analytical procedure could be automated and the amount of labour significantly reduced. Even the environmental impact of the analytical procedure itself is reduced through use of much smaller amounts of solvents. This set-up was used in the work reported in Paper I.

In the LAQUA protocol the analyses of organic compounds are divided to address three main parts: polar, non-polar and volatile organic compounds. In each part, specific marker substances have been selected for monitoring.

Polar organic compounds are an important group when monitoring organic pollutants in leachate. In the suggested protocol, different specific phenolic compounds are selected as markers for this group of contaminants, due to their known presence in leachate and their high environmental impact. Five different phenols found to be commonly present in leachate in preliminary experiments were chosen as representative markers. These were phenol, p-cresol, o-cresol, 2,4-dimethylphenol and 3-methyl-4-chlorophenol. 4-chlorophenol was chosen as a surrogate standard. A mixture of these six phenolic compounds was used for the optimisation of the extraction system and the final analytical procedure. The automated analytical system, using SLM extraction coupled on-line to HPLC with DAD as mentioned above, is thoroughly described in Paper I.

A common method for determination of phenols in wastewater is the phenol index method. This method is referred to in the waste acceptance criteria [18, 19]. The phenol index method measures the sum of distillable phenols that react with 4-aminoantipyrine. However, 4-aminoantipyrine reacts poorly with several para-substitued phenols. It is stated that 4-aminoantipyrine does not react with either 2,4-dimethylphenol or p-cresol [121], which are both found in the leachates studied within this thesis. In fact, p-cresol had the highest concentration of any of the identified phenolic compounds in the studied raw leachates. In the study of leachability of sludge from stormwater drainage wells (Paper V), analyses of the same samples were performed with the phenol index method and with the described method in the LAQUA protocol. The comparison showed that a determination using the phenol index method markedly underestimated the concentrations of phenolic compounds in the eluate water. Thus, using the phenol index for risk evaluation of landfill leachate seems to be questionable.

Just monitoring the sum of phenolic compounds does not give full insight in the behaviour of a treatment procedure. This can be seen Figure 2 (reprinted from Paper I).

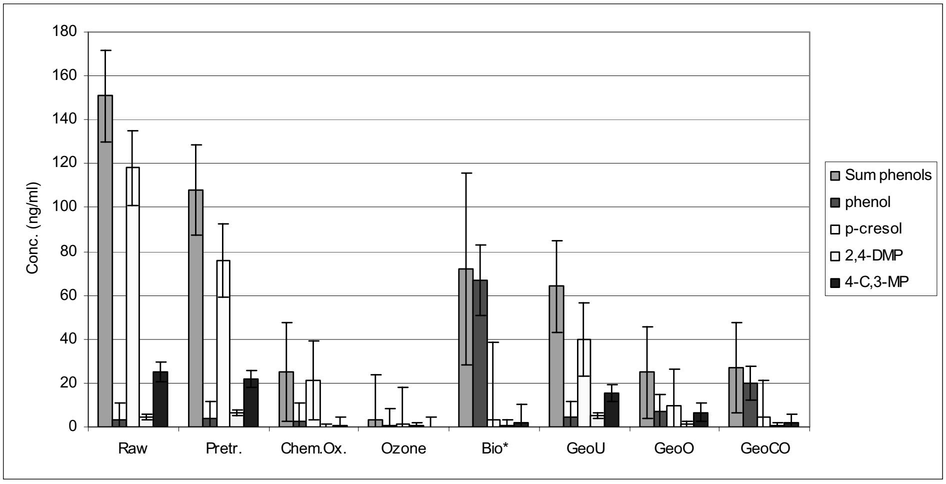


Figure 2*. Results for sum of phenols, and for four major identified phenols. The error bars show the 95 % confidence intervals for the average (n = 8 except for bio where n = 2) of samples, obtained from time integrated sampling on different dates*

From the bioremediation step (Bio.) it is clear that all phenolic compounds were efficiently removed except for phenol itself, which instead dramatically increased compared to the pre-treatment step (Pretr.). This shows that the degradation of some other phenolic compounds most probably give phenol as the final product.

Non-polar organic compounds

Most of the known POPs are non-polar compounds, and monitoring these is of great interest in any type of wastewater. The classical POPs as PCBs and PBDEs, were selected as markers for non-polar organic compounds in the evaluation of the treatment efficiency of leachate (see Paper II). Identification and quantification was performed on 29 PCB congeners, with degree of chlorination varying from 1 to 10 chlorine atoms, and of two PBDEs (one hexa-BDE and one hepta-BDE).

Analysing organic compounds generally requires some form of pre-treatment of the sample. This sample preparation may include steps like filtration, pH adjustment, a variety of extraction procedures, analyte trapping, etc. Concentrations of the interesting organic compounds are usually very low in leachate samples, and the goal of the sample preparation will thus be both clean-up and enrichment of the analytes. There are drawbacks with the classical extraction methods for non-polar organic compounds, as previously mentioned. The major ones are the large amounts of solvents used and the considerable manual work required. Therefore, within the study presented in Paper I, the analytical procedure used was based on SPE followed by automated supercritical fluid extraction (SFE) to elute PCBs and PBDEs from the SPE discs, which were used in the first sampling step.

Volatile organic compounds

Volatile organic compounds (VOCs) are a large group of anthropogenic or biogenic organic compounds with relatively high vapour pressures. Within the EU, VOCs are defined as any organic compounds having a vapour pressure of 0.01 kPa or more at 20ºC [122]. The composition of VOCs from landfills has been widely studied due to their toxic nature and adverse environmental impact (unpleasant odours, poor air quality, health problems). VOCs emitted from municipal waste are typically generated at various stages in the decomposition of organic substances under anaerobic as well as aerobic conditions. Some of the emitted VOCs are hazardous, *e.g.* benzene has carcinogenic properties. The occurrence of benzene, toluene ethylbenzene and xylene (BTEX) in the waste can be attributed to dyes, pesticides, solvents in paint, paint, etc. [123].

VOCs can be found in water and in air, and consequently there are various techniques to determine them according to the medium they are in. A method for collection of VOCs in air is to use impingers containing a solvent. Another common methodology is sampling using adsorbent tubes and subsequent release of the analytes with either an organic solvent or by thermal desorption. In the latter case automated equipment is often used with GC-MS as the final determination step [124]. Over 150 compounds have been identified and the 30 most abundant ones were quantified in a study of waste disposal bins by Statheropoulos *et al* [123]. In a study by Nammari *et al.* [125] on emissions to air from baled municipal waste, a list of 240 compounds was compiled from a literature survey of common VOCs emission from waste sources.

5.1.3. *Artemia salina* for assessment of acute toxicity in leachate

The amount of chemical substances present in leachate is very high, and water-purity classification based entirely on physical and chemical parameters is not sufficient to estimate the risk associated with chemical pollution of the water in a given aquatic environment. Accordingly, a toxicity test for screening of total acute toxicity was developed in the LAQUA project (see Paper III). The goal was that the toxicity test should give results in a short time, that it would not require the use of any specialized costly equipment, and that it should work for different types of landfill leachates. Since leachate from municipal landfills often contains high concentrations of chloride and ammonia, where the salinity may influence the toxicity for fresh water organisms, the salt-durable crustacean *Artemia salina* was investigated for assessing leachate toxicity. A test using the brackish water organism *Artemia* is available on the market as Artoxkit [126] and the organism is well known to be easy to handle and the method is known to give good repeatability [127, 128]. This method is standardized and described by the Artemia Reference Centre (ARC), Gent, Belgium [129]. With the ARC-test in mind, the methodology for acute toxicity testing of leachate was developed and tested for leachate treatment procedures.

The testing procedure developed is easy to perform and the equipment is available in most laboratories. Figure 3 shows the equipment used for the test. The procedure is partly described below and in more detailed in Paper III.

***Figure 3**. Equipment for hatching the Artemia larvae (left) and procedure for preparing the samples and scoring the results in the acute toxicity test (right).*

One advantage with toxicity tests using crustaceans such as *Artemia* is that the organism is available in batches of “dormant eggs” (cysts) and a new population of the organisms is hatched for each test. This will ensure the reproducibility of the test and assure that the whole tested population is in the same condition. The *Artemia* cysts used in the methodology described in Paper III are easily, and cheaply and accessible in pet shops, where they are sold as food for aquarium fish. The hatching is performed in a standard artificial seawater of a salinity concentration of 35 g/L, prepared according to Dietrich & Kalle [130]. After 24 hours at 25ºC, the hatched larvae at first instar will be transferred to fresh hatching medium, and the larvae will moult into the next and most sensitive instar within another 24 hours. Hence, the hatching of the larvae for the toxicity test will take 48 hours.

The toxicity test is performed on multi well plates (Nunclon^{TM} Multidish 48) with a sample volume of 400µl in each well. The leachate is diluted, according to the critical range of the actual leachate obtained during prestudies with the organism. The range mainly used for *Artemia* tests within this thesis was 45% to 91% leachate dilution. Each concentration is duplicated in three wells on the plate. From the hatching dish, 40µl of hatching medium with about 5 to 20 *Artemia* larvae is collected and transferred to each well on the multi dish plate. After incubating at 25ºC at 24 hours the plate is placed under a dissection microscope and the numbers of immobile larvae are recorded. The hatching medium (standard artificial seawater) is used as a negative control, and a reference toxicant, potassium dichromate ($K_2Cr_2O_7$) is used as a positive control.

One disadvantage with this toxicity test method is that the counting of the immobile larvae has to be done by ocular determination. To decide if the larvae are immobile (*i.e.* don't move forward for 10 seconds) can sometimes be difficult. However, the reproducibility of the test is high. *Artemia* tests have been performed several times by students attending an environmental chemistry course, and the results of the reference toxicant have been within the range determined during the development of the test.

Figure 4 (reprinted from Paper III) shows how the immobility of *Artemia salina* depends on ammonium and ammonia concentrations in different media.

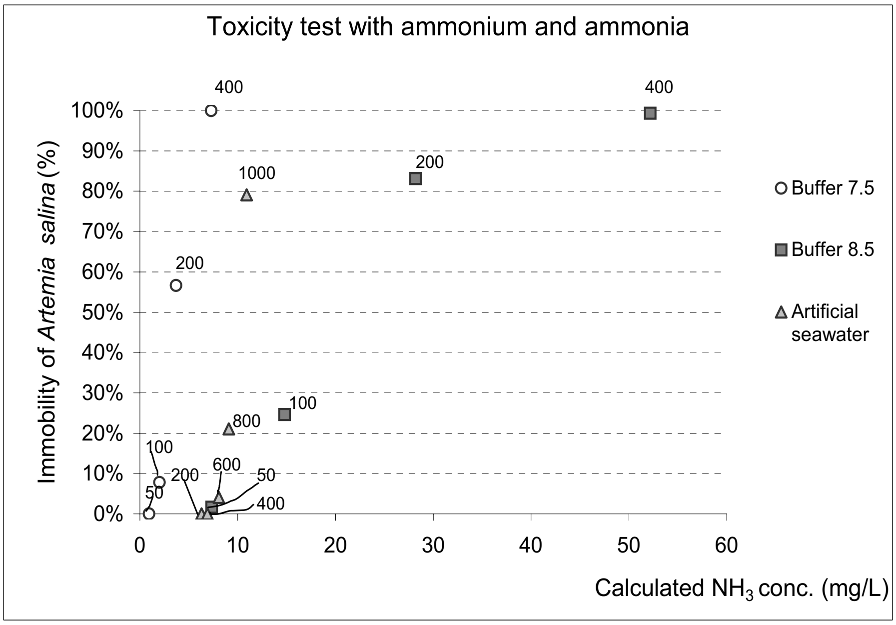


***Figure 4**. Immobility in % of Artemia salina incubated for 24h in three different solutions of ammonium chloride diluted in buffer 7.5 (HEPES and NaOH); buffer 8.5 (Borax and HCl); and in standard artificial seawater, plotted against calculated concentration of ammonia (mg/l). Numbers in the plot area are concentrations of ammonium in mg/l.*

For each of the three solutions shown in Figure 4, it is clear that the immobility increases when the ammonia concentration increases. Thus, the concentration of ammonia is the main factor for toxicity measured by the *Artemia salina* test. The sensitivity of *Artemia salina* in the two buffers differs considerably for a certain ammonia concentration, which implies that the overall composition of the solutions also has a strong impact on the results. As an example, an ammonium concentration of 800 mg/l in artificial seawater (pH 7.6) gives only about 20% immobility compared to the Hepes buffer at pH 7.5, for which the immobility was 100% already at 400 mg/l ammonium, even though the ammonia concentrations were almost identical (about 7 mg/l). Thus, the influence of the composition of the chosen medium has a large impact on the results, and with present knowledge artificial seawater is considered to be the best dilution medium. The tolerance towards ammonium was however shown to be relatively high. When the test solution with ammonium was diluted in artificial seawater, less than 10% of the organisms in the population became immobile in an ammonium concentration of 600 mg/l. Furthermore, the toxicity of the leachate disappeared after passage through ion-exchange columns where ions such as ammonium and metals were removed. When the leachate had percolated through a column with active carbon – a method to keep the ammonium and metal concentrations constant but to remove most organic compounds – the toxicity was still relatively high (Figure 3 in Paper III). Tests with simple solutions of individual heavy metals showed that the toxicity of heavy metals was low towards *Artemia,* which indicates that most of the acute toxicity in landfill leachate is associated with ammonia/ammonium. This is further supported by the results of the treatment procedures, tested in the pilot plant study, for which decreasing the ammonia/ammonium concentration led to significant reduction in toxicity (Figure 1 Paper III). A correlation between the amount of non-ionized ammonia and toxicity has also been shown by other authors [45, 102, 113, 131].

Several authors have described the possibility of fractionation of the sample in order to identify the toxic agents in water [94-96, 103]. In our study fractionation of the leachate was made using columns containing ion-exchange resins and activated carbon. These fractionations gave significant amounts of extra information that was useful in assessing the environmental impact of different constituents in the leachate. The toxicity test with *Artemia salina* has been shown to be a useful complimentary tool for evaluating the different treatment techniques used in the pilot plant study, as described in Paper II. In accordance with this, in a study by Silva *et al.* [132], *Artemia salina* was shown to be an organism that gave toxic responses in the tested treatment procedures and could thus be generally useful for evaluation of treatment methods for leachate. In summary, ammonia and ammonium seems to be responsible for most of the toxicity in leachate as supported by several findings in this study.

5.2. Application of the LAQUA protocol

The analytical protocol was applied in the evaluation of the efficiency of different treatment methods in a pilot plant for local treatment of leachate from Härlöv landfill, the MSW deposit situated in Kristianstad, Sweden (described in Paper II). The same protocol was used for evaluation of treatment efficiency in a lab-scale column study of different filter materials on leachate from an industrial landfill for fragmented metallic waste residuals at Halmstad, Sweden (described in Paper VI).

5.2.1. Pilot plant study

A pilot plant for evaluation of different leachate treatment methods was constructed within the LAQUA project, at Kristianstad MSW landfill site (Paper II). The deposit is situated in “Kristianstads Vattenrike”, a marshland which year 2005 became the first biosphere area in Sweden through UNESCOs ”Man and the Biosphere Programme”. The landfill was in use from the beginning of year 1960 until 2002, and for the moment top-covering of the landfill is ongoing. A drainage system of liners at the bottom of the landfill and ditches collects the leachate. The leachate is pumped to the municipal wastewater treatment plant, and is thus treated together with sewage. The average volume of leachate generated for the years 2003 to 2007 was about 300 000 m^3 a year, with an increase during the latest years [133]. There are problems with mixing leachate and sewage in a treatment plant, and the local authorities in Kristianstad attempt to find other solutions for landfill leachate treatment. Hence, within the LAQUA project, a pilot plant for evaluating different treatment methods was built on the actual landfill site and this study is presented in Paper II.

The pilot plant was constructed as a flow-through system (Figure 1 in Paper II), where the raw leachate was pre-treated by aeration combined with sedimentation, and then by one of four different treatment procedures: bioremediation, ozonation, chemical oxidation by Fenton’s reagent, or geo-bed filters. These techniques were chosen due to known capacities in reduction of organic pollutants in wastewater, and operated with the best management procedures according to their suppliers (Paper II).

Samples were obtained weekly through a time-integrated sampling method, where intermittent pumping to temporary collection vessels was used. Aliquots from these vessels were collected on a daily basis and stored in a refrigerator before analysis.

Time-integrate sampling is necessary in order to compensate for the possible variations of the inflow to the treatment plant. Evaluation was performed according to the LAQUA protocol, and the composition of the raw leachate is shown in Table 1, section 2.3. The results from the analysis of the chosen sum parameters, metals and organic compounds are shown in Table 4 (reprinted from Paper II).

Table 4. *Average values based on 3-8 determinations of chosen parameters before and after treatment of leachate. Relative standard deviation (RSD) in % is given within parenthesis.*

Parameter		**Raw**	**Pretr.**	**Chem Ox**	**Ozone**	**Bio**	**GeoU**	**GeoO**	**CeoCO**
Metals									
Zinc, Zn	μg l^{-1}	60 (24)	51 (21)	84 (12)	72 (26)	425 (5)	55 (24)	55 (15)	60 (19)
Chromium, Cr	μg l^{-1}	15 (27)	11 (20)	9 (33)	10 (22)	15 (38)	10 (17)	11 (37)	7 (45)
Copper, Cu	μg l^{-1}	5 (29)	5 (38)	10 (49)	25 (28)	10 (17)	9 (62)	5 (38)	9 (32)
Lead, Pb	μg l^{-1}	2.2 (28)	1.7 (4)	1.1 (26)	1.7 (17)	1.9 (27)	3.0 (30)	3.4 (34)	2.1 (22)
Cadmium, Cd	μg l^{-1}	0.2 (75)	0.2 (73)	0.4 (65)	0.2 (68)	0.4 (46)	0.5 (58)	0.4 (40)	0.5 (31)
Arsenic, As	μg l^{-1}	5.9 (10)	5.3 (16)	3.6 (52)	5.2 (19)	6.2 (6)	6.2 (26)	5.6 (21)	15 (12)
Organic compounds									
Phenolic compounds (sum of the 4 comp. below)	μg l^{-1}	151 (27)	108 (57)	25 (112)	2.9 (76)	72 (40)	64 (29)	25 (18)	27 (81)
Phenol	μg l^{-1}	3.5 (72)	3.8 (45)	2.8 (142)	0.6 (283)	67 (38)	4.3 (73)	7.2 (48)	20 (125)
Paracresol	μg l^{-1}	118 (24)	76 (73)	21 (105)	1.5 (53)	3.1 (26)	40 (36)	9.9 (35)	4.3 (34)
2,4 methyl phenol	μg l^{-1}	4 (75)	6.2 (28)	<0.5	0.6 (263)	<0.5	5.1 (28)	1 (110)	<0.5
4-chloro, 3-methyl phenol	μg l^{-1}	25 (47)	22 (31)	0.5 (265)	0.3 (283)	1.4 (141)	15 (34)	6.7 (42)	2.1 (15)
PCBs (sum of 28 congeners)	ng l^{-1}	13 (22)	8.6 (52)	4.9 (67)	3.4 (50)	8.4 (19)	5.5 (43)	6.9 (74)	6.8 (76)
BDE-153	ng l^{-1}	14 (69)	16 (69)	11 (118)	13 (78)	s.e.	28 (85)	12 (71)	14 (70)
BDE-183	ng l^{-1}	107 (43)	91 (45)	42 (51)	58 (62)	63 (37)	92 (71)	55 (34)	60 (45)
Unidentified non-polar organic compounds (sum of 10 peaks, normalised)	%	100	52 (85)	13 (84)	20 (47)	19 (128)	24 (94)	26 (129)	17 (130)
Sum parameters									
pH		7.1 (3)	8.6 (2)	7.6 (9)	8.0 (3)	7.7 (2)	7.5 (7)	7.8 (4)	7.6 (2)
Conductivity, 25°C	mS m^{-1}	729 (3)	632 (2)	781 (5)	629 (2)	544 (7)	599 (6)	607 (3)	629 (3)
BOD$_7$	mg l^{-1}	29 (33)	14 (58)	31 (23)	29 (44)	s.e.	24 (47)	10 (40)	5 (56)
TOC	mg l^{-1}	128 (10)	108 (12)	90 (17)	57 (26)	94 (9)	198 (22)	129 (28)	64 (12)
NO$_3$-N	mg l^{-1}	3.2 (29)	2.2 (39)	4.4 (18)	24 (32)	150 (27)	<0.2	11 (39)	68 (30)
NH$_4$-N	mg l^{-1}	236 (5)	227 (8)	211 (11)	201 (13)	5 (76)	215 (1)	182 (24)	108 (23)
Total-N	mg l^{-1}	246 (12)	231 (11)	224 (6)	228 (5)	175 (13)	223 (14)	218 (10)	198 (16)

s.e. sampling error

Almost all of the evaluated parameter values and compound concentrations decreased in the pre-treatment procedure. Hence, a simple pre-treatment with aeration and sedimentation should always be included in a treatment plant. Chemical oxidation and ozone-treatment gave the best reduction of all kind of organic compounds, but was associated with only small changes in other parameters. In the bioremediation step it is clear that all phenolic compounds are efficiently removed except for phenol itself, which is dramatically increased compared to the pre-treatment step. For degradation of phenolic compounds from wastewaters, the establishment of a microbiological environment, including both fungi and bacteria, is required [134]. It is evident that bioremediation is very efficient for removal of most of the phenols, but an extra refinement step or longer remediation time is needed before discharge of the effluent into the recipient. The analysis of the nitrogen compounds shows that the bioremediation step reduced almost all the ammonium, and the corresponding nitrate increased. The results after each geo-filter step show that the filter bed consisting of a mixture of peat and carbon-containing ash (GeoCO) gave the best reduction of organic compounds, and the removal is as good as in the oxidation and bioremediation methods. Of note was the decrease of BOD and TOC in the peat and carbon ash filter, which might be related to biological activity in a biofilm, which was obvious also when considering the decrease of ammonium and increase of nitrate.

The results from the acute toxicity tests with *Artemia salina* are shown in Figure 5 (reprinted from Paper III).

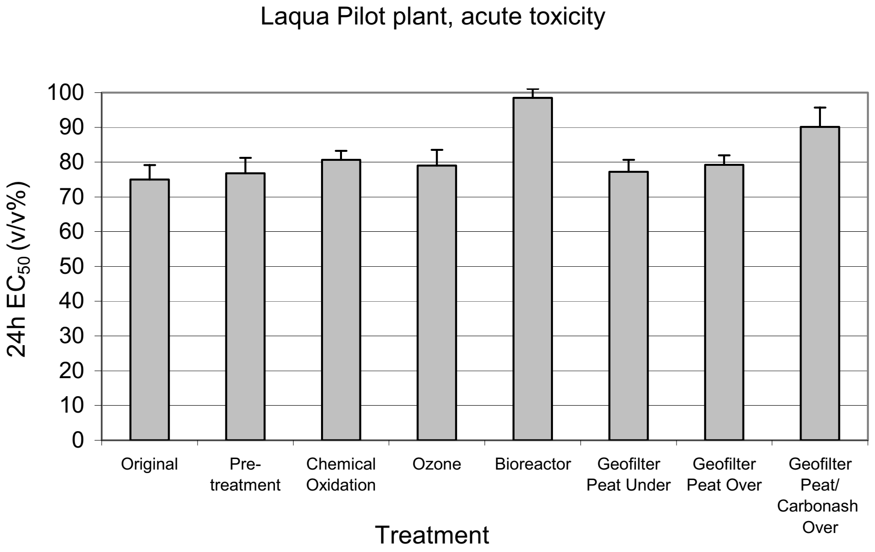


Figure 5. *Determined acute toxicity in MSW landfill leachate after different treatment procedures, studied in a pilot plant. Average 24h* EC_{50} *values for Artemia salina (n=4-10), with bars showing the one-way standard deviation.*

It can be seen that the toxicity in the raw leachate is almost eliminated after passing the bioreactor. Also after passing the geo-filter containing carbon ash, the leachate is considerably less toxic than after the pre-treatment. In both cases there is a large decrease of ammonium/ammonia in the leachate. Thus, in treatment procedures with high transformation of the nitrogen compounds from NH_3 / NH_4^+ to NO_3^-, the effluent water will be associated with lower acute toxicity.

The concentration of zinc increased markedly in the bioreactor, and copper increased in the ozone, chemical oxidation and bioremediation steps, all these changes probably being due to the release of these substances from the tank constructions. However, the metal values were still very low in the leachate, and should not significantly influence the results.

The study showed that one obtains a good evaluation of what happens during different treatment procedures by measuring the analytical parameters specified in the LAQUA protocol. The chosen marker compounds and the biomarker give a good overall picture of changes in the concentrations of pollutants, which is valuable when choosing a treatment method for the actual landfill leachate. A combination of pre-treatment with a main treatment step based on a filter bed with peat and carbon-containing ash, seems to be good economical choice for leachate treatment.

5.2.2. Column study

A column study for investigation of treatment efficiency of different filter materials on leachate from an industrial landfill for fragmented metallic waste residuals at Halmstad, Sweden, was set up in a laboratory and is described in Paper VI. The LAQUA protocol was applied in the study. The investigation was focused on the efficiency of removal of dissolved organic carbon (DOC), polar and non-polar marker compounds, and metals. The objective was to determine whether a selected mixture of substances included in the filter medium might be able to simultaneously trap both inorganic ions and organic compounds with widely differing properties.

The filter materials chosen are all either abundant in nature, by-products or waste products from industry, or products from agricultural operations. Some materials were chosen based on previous experiments, *e.g.* the peat and carbon ash mixture (described in Paper II). The main component in the mixtures has been peat. Peat has been tested alone but also mixed with carbon-containing ash, polyurethane (PUR) and wood pellets. A detailed description of the filter mixtures can be found in Paper VI.

The experimental set-up consisted of columns packed with these selected materials (see Figure 1 in Paper VI). In short, the raw leachate (collected at the actual landfill) was stored and pre-treated by aeration in a tank (1 m^3), and transferred to smaller containers. The pre-treated leachate was thereafter pumped onto the columns (downwards flow mode) for 60 min twice a day at a flow rate of 9 mL/min. Time-integrated sampling of the effluent was obtained by continuous collection of each effluent for 24 hours. Eight samples were taken during a test period of six months. Grab samples of the influent were taken at the same time as the effluent sampling started.

As stated before, the LAQUA protocol should be seen as a dynamic protocol, and parameters for evaluation may be changed in accordance with the actual wastewater being considered and the purpose of the investigation. Since the actual industrial landfill leachate contains higher concentrations of metals than MSW landfill leachate, a higher number of metals were included in the column study compared to the ones measured in the MSW landfill leachate (described in Papers I-III). The acute toxicity test with *Artemia salina* was excluded in this column study, due to results from previous tests indicating that the actual raw leachate was not toxic to *Artemia salina* [47].

The results of the column study showed that the mixture of peat and carbon-containing ash gave the most efficient removal of different metals and organic pollutants compared to the other materials. This mixture was thus recommended to be tested further as a filter material in a treatment plant based on the biofilter technique. Pilot plant and full-scale plant studies using this mixture are described by Kängsepp *et al.* [47, 135, 136].

5.3. Investigation of the performance and application of leaching tests

In these investigations, described in Papers IV, V and VII, different waste materials were characterized according to their leachability by using two EU standardised tests for leaching of waste. The up-flow percolation test SIS-CEN/TS 14405:2004 [75], and the batch test SS-EN 12457 [74] were further investigated towards new pollutants not presently described in waste acceptance criteria (WAC) and towards the use of a leachant other than demineralised water (Paper IV). Paper IV and Paper V describe how the leaching tests were used to characterise and evaluate the hazards of fragmented metallic waste and sludge from stormwater drainage wells, respectively. Paper VII presents a methodology based on batch-tests, for evaluation of a filter material aimed at leachate treatment. The filter material, a mixture of peat and carbon-containing ash, was characterised according to its efficiency as a sorbent and its leakage of different substances from the material, before and after usage.

5.3.1. Leaching tests of metallic waste

When the investigation described in Paper IV was performed, the two leaching test methods were not yet approved and the scientific experience with the tests was limited. One aim of the study was to evaluate the leaching of two different types of fragmented metallic wastes using the LAQUA analytical protocol, in order to extend the analyses of organic parameters. Furthermore, in order to investigate the natural scenario in a landfill, namely the percolation of leachate through the waste body, the use of leachant was extended with landfill leachate besides the prescribed use of demineralised water. The two leaching methods were also compared with respect to their leaching strength and procedure performance.

In order to obtain representative samples of waste material, which is normally highly heterogeneous, a standardized sampling procedure is very important [75]. The sampling procedure of the two fractions of fragmented material was carried out according to a Nordic standard [137], wherein the samples were further divided in several described steps down to test samples of 100 g dry weight for the batch test, and approximately 1 kg dry weight for the percolation test.

The equipment, materials and procedures for the two test methods are described in detail in Paper IV, and are shown in Figure 6. The two methods differ considerably in equipment, amount of labour and time needed.

***Figure 6.** Experimental set-up and equipment used in the up-flow percolation test (left) and the shake-batch test (right)*

In brief, the percolation test was performed in plastic columns with a diameter of 10 cm and a height of 30 cm filled with material. Filters of glass wool and sand were placed in the inlet and outlet of the column. The leachant was percolated through the column with an upward flow set by a peristaltic pump delivering the leachant at a continuous flow rate of 48 ml/h. In a percolation test it is easy to collect several different L/S fractions, and in this study three different fractions (L/S 0.1, 2 and 10) were collected separately. The time needed to obtain the L/S 10 fraction was approximately one month. The batch test was performed as a two-stage shaking test. In the first leaching test (giving fraction L/S 2), waste material and leachant were agitated together for 6 hours and the eluate was separated from the solid through filtration. In the second leaching step (giving fraction L/S 8), new leachant was added to the solid material and the bottles were agitated for a further 18 hours. The time needed to perform the batch test was about two days. After analyses of different parameters in the different eluate fractions, calculations were made, according to the descriptions in the standards, to obtain cumulative leaching values for each parameter, expressed in mg/kg dry weight.

Results on analyses of organic compounds in eluates showed that it was possible to use these test methods to evaluate the leaching of such compounds from waste materials. Phenolic compounds and PCBs were found in eluates from both types of tests and in both materials. The analyses of six different phenolic compounds were performed using HPLC and the phenols were identified from spectra and retention times of standards. One typical chromatogram from an analysis of phenolic compounds is shown in Figure 7 (reprinted from Paper IV).

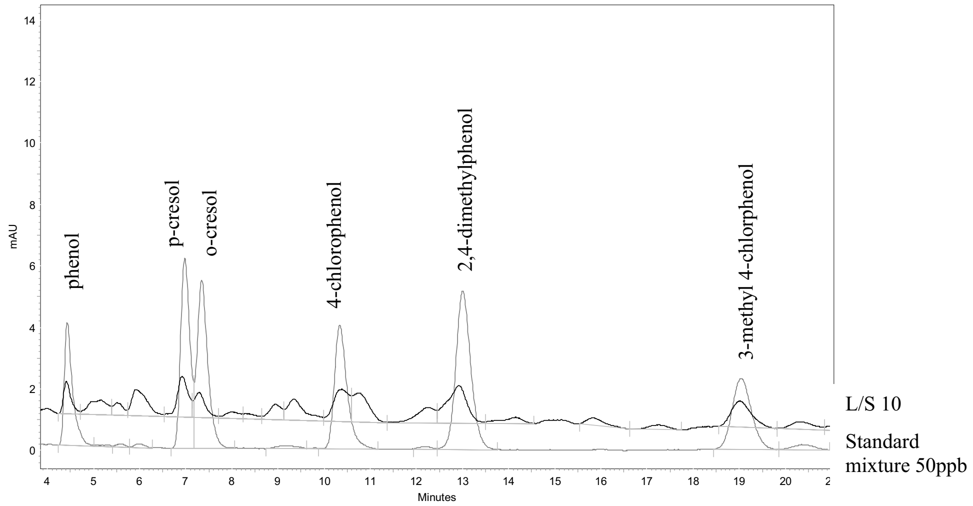


Figure 7. *A typical chromatogram from an analysis of phenolic compounds in wastewater using HPLC-DAD with on-line SLM extraction. The sample was the collected eluate fraction L/S 10 in a percolation test of fragmented metallic waste. A chromatogram obtained from analysis of a standard mixture of 50 ppb of 6 phenolic compounds is also shown.*

The main problem in this case is to find a methodology for, analysing at a reasonable cost, the large number of organic compounds which almost certainly will be found in the eluates. This will probably lead to a need to establish a limited number of markers, which can be quantified and which will build a base for the decision about the waste category appropriate for the given sample.

In the eluate from percolation tests using leachate as leachant, most of the parameters have higher concentrations compared to the concentrations obtained using demineralised water. As can be seen in Table 5 in Paper IV, the differences are in several cases large. This is evident in the fraction L/S 10, for which the concentrations of Cu, Fe and Ni in demineralised water were 1.3, 2.1 and 0.16 mg/kg respectively, compared to concentrations of 20.9, 7.7 and 1.1 mg/kg, respectively, using leachate water as leachant. On the other hand, the concentrations of non-polar organic compounds tend to be lower using leachate water as leachant. In the percolation test it was also noticed that the eluate concentrations for some parameters (DOC, sulphate and phenolic compounds) were lower than in the ingoing leachate. This was not noticed in the batch test, and could have been due to adsorption/desorption and/or redox reactions in the percolation column during the relatively long time it takes to perform this test compared to the considerably shorter time needed for running a batch test. The study has certainly shown that the use of landfill leachate water as leachant, leads to significantly increased concentrations of several heavy metals. However, the uncertainty about the composition of the leachate, could for the sake of simplicity, favour a well-defined alternative such as *e.g.* demineralised water as stated in the test methods. On the other hand, a choice of a synthetic leachant containing salts and/or ammonia would give a more conservative estimate of the risk of environmental hazards.

The batch test was performed in five replicates and the percolation test was performed in three replicates on each material and the precision of the tests was measured by the relative standard deviations (RSD) of the analysed parameters. Even if the sampling procedure was very accurately done, the heterogeneity in the waste material is obvious from the high RSD values. On average the RSD was 48% for the batch test and 35% for the percolation test. This points towards a large inhomogeneity of the tested materials, since the uncertainty in the final analysis step is expected to contribute only by a few percent to the total RSD value. Many of the metals showed RSD values over 100% and this must be taken into consideration in a risk assessment. This will lead to difficult decisions about whether a result is acceptable or not, and whether a single value can be considered as an outlier. Thus, to be able to make a valid risk evaluation of this type of material, it is important to carry out a sufficient number of replicate measurements in each test to improve the accuracy of the average value.

5.3.2. Leaching tests of sludge from stormwater drainage wells

The purpose of the investigation of sludge from stormwater drainage wells was to gain more knowledge about the leaching test methods to be able to make future decision on the general applicability of the proposed tests. The analyses of the obtained eluates were extended by further investigations of the organic part, including volatile organic compounds (VOC) and semi volatile organic compounds (SVOC). The sampling procedure, the material and the two leaching tests, a two-stage batch and an up-flow percolation test, are described in detail in Paper V. In this investigation the analyses of the eluates were performed on a mixed collected eluate sample based on five replicates in the batch tests and duplicate eluates in the percolation tests.

The analyses of the volatile organic part, the gasoline compounds benzene, toluene, ethyl benzene and m-xylene (BTEX), gave generally low concentration values. Thus, there seems in general not to be any problems concerning concentrations of volatile aromatic substances in sludge samples. These substances apparently are so volatile that most of them disappear by evaporation from the gutters or during the dewatering step. Concentrations of SVOCs from analyses of sludge, batch eluate and percolation eluate are collected in Table 5.

Table 5. *Retention times (RT) and concentrations (µg kg^{-1} DW) of organic compounds in sludge from streets with low traffic intensity (LT) and high traffic intensity (HT) and in corresponding eluates, fraction L/S=2 l kg^{-1}, from percolation and batch test (reprinted from Paper V).*

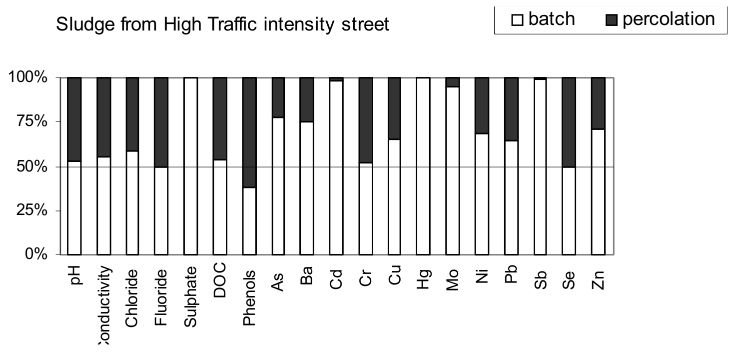
Compound (µg kg-1 DW)	RT (min)	Sludge		Percolation test		Batch test	
		LT	HT	LT	HT	LT	HT
Benzothiazole	14.33	280	530	1	2	5	2
Tributyl phosphate	17.72	60	45	0.4	0.7	0.6	0.7
Butylated hydroxytoluene	17.78	26	25	0.1	0.4	0.5	0.5
Ditertbutyl phenol	17.87	18	27	3	0.6	0.3	0.6
Diethyl phthalate	18.91	110	100	5	14	11	15
Butyl benzene sulphonamide	21.34	6	2	0.6	0.3	0.6	0.3
Dibutyl phthalate	22.90	560	250	3	9	7	9
Alkyl biphenyl	24.47	1110	610	0.04	0.5	0.6	0.5
Pyrene	25.05	1700	1710	0.04	0.7	0.3	0.8
Triphenyl phosphate	27.03	<0.1	<0.1	1	0.1	2	0.1

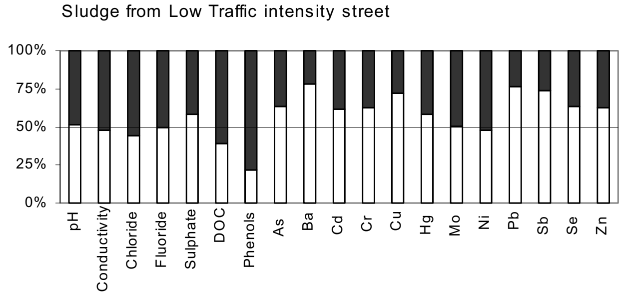
The concentrations are similar in sludge, formed at high or low traffic intensity, with pyrene as the dominating compound. As expected, the same types of substances were found in the eluates as in the sludge, with the concentrations in the eluates of all the considered pollutants in the low ppb range. The concentrations of these compounds in the sludge are in general 10-1000 times higher. Many of the compounds in Table 5 have previously been found in leachate from landfills [42, 46, 138].

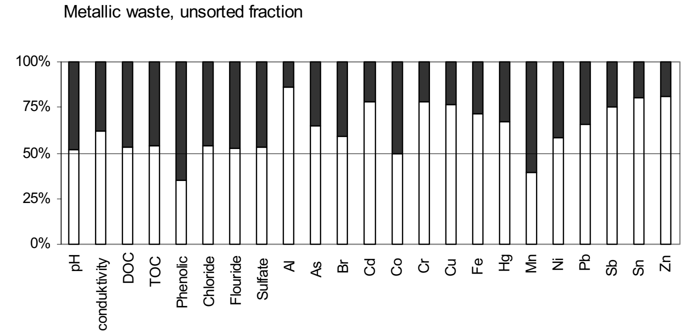
A weakness in the present tests and WAC from non-hazardous and hazardous waste is that the possible toxicological impact of persistent organic pollutants is greatly neglected. To ascertain that the concentrations of highly toxic compounds are low, a GC-MS screening and possibly also toxicity tests should be included in the risk assessment. However, the acute toxicity test with the brackish water organism *Artemia salina* that is included in the LAQUA protocol may not to be sensitive enough for toxicity investigations of this kind of non-hazardous eluate from leaching tests. The acute toxicity tests with *Artemia* performed on the eluates from leaching tests in this investigation and on the eluates from metallic wastes (Paper IV), showed no immobility of the organism in any of the eluates obtained with de-mineralized water as leachant. Thus no toxicological values could be obtained. To be sure that the eluates from the investigated materials were not acutely toxic, toxicity tests with other organisms should preferably be considered.

The phenol index, proposed in the leaching test procedures for inert waste, will give some information about the toxicity of polar organic compounds in the eluate. However, it seems that the actual phenolic concentration is significantly underestimated. A better approach would most probably be to use HPLC for determination of the concentration for dominating phenolic compounds, as exemplified in this study.

The batch test is a compliance test and the percolation test is a characterization test and they certainly have different purposes, but one feature to consider is whether they give similar results. A percolation test for characterization of waste takes a long time to perform (ca. 30 days). A batch test will be performed in a much shorter time (2-3 days), and it would thus be advantageous if a decision about deposition of a waste could be based on a batch test. Then it is important that this test gives a conservative estimate of the situation, *i.e.* give values that are not lower than values that might be obtained with a percolation test. A comparison is made in Figure 8, where the contribution ratio of the sum of results from batch test and percolation test are given for the investigated parameters.







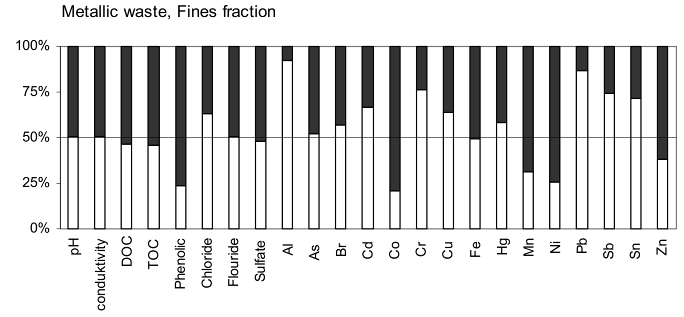
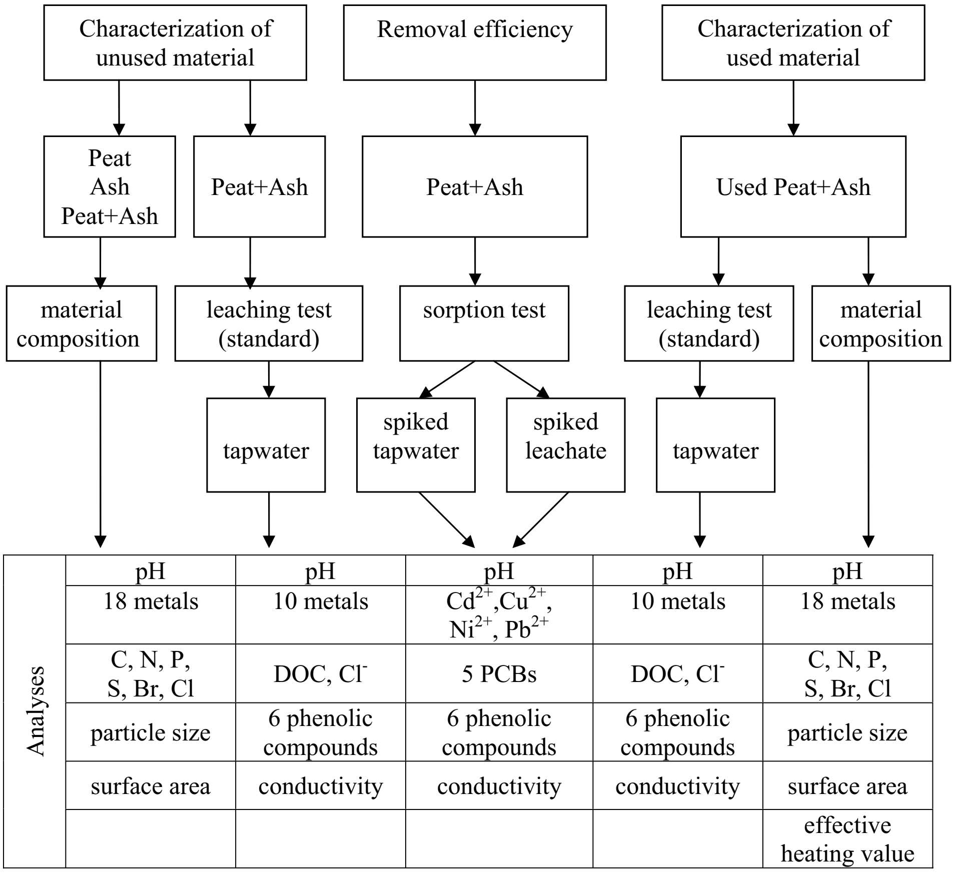


Figure 8. *Relation between leaching values (fraction L/S 10) obtained by batch test □ and percolation test ■, of four different waste materials. The contributions in % are obtained from the sum of batch and percolation values for each parameter.*

The results of the comparison for the materials investigated in this study and in the study of fragmented metallic wastes (presented in Paper IV) show that the values from the batch test generally are higher with a few exceptions. Other authors have presented similar results [63, 70]. Due to the generally good agreement found between the results of the two leaching methods, the batch test can be seen as an alternative to the percolation test. It can also be noticed that the result from the two-stage batch test at accumulated L/S 10 can be said to be more similar to the percolation test, because the constituents leached in the first stage are withdrawn and a new chemical equilibrium can develop in the second stage [139].

5.3.3. Strategy for investigation of a filter material for leachate treatment

An investigation of a geological filter material, based on a mixture of peat and carbon-containing ash, and aimed at leachate treatment is described in Paper VII. The presented strategy is based on batch equilibrium experiments and includes three main parts. First, a characterization of the filter material, next an investigation of the removal efficiency of different pollutants from the material, and finally a characterization of the filter material after long-term use. Since the aim was that the final outcome should be the manufacture of an environmentally friendly adsorbent, the chosen materials were naturally abundant and a waste residual. A filter material needs to be exchanged occasionally either because it has been saturated by pollutants or because the hydraulic conductivity has decreased too much. Two possible alternatives for further handling of the material, landfilling or incineration, can be selected. To determinate whether the material can be characterized as non-hazardous waste, and thus suitable for deposit in a landfill for such waste, a standardized leaching test can be performed. Incineration, the favoured handling of these two alternatives, according to the hierarchy of waste [12], may be preferred if the heating value is high enough and the metal content is low [140]. The strategy for investigation of the filter material is shown in Figure 9 (reprinted from Paper VII).



Analyses	pH	pH	pH	pH	pH
	18 metals	10 metals	Cd^{2+},Cu^{2+}, Ni^{2+}, Pb^{2+}	10 metals	18 metals
	C, N, P, S, Br, Cl	DOC, Cl^-	5 PCBs	DOC, Cl^-	C, N, P, S, Br, Cl
	particle size	6 phenolic compounds	6 phenolic compounds	6 phenolic compounds	particle size
	surface area	conductivity	conductivity	conductivity	surface area
					effective heating value

Figure 9. *Outline of the strategy for investigation of a filter-material aimed for leachate treatment.*

The filter material, a mixture of peat and carbon-containing ash (Peat +Ash) with a volume ratio of 3:1, was investigated as well as its components (Peat) and (Ash). The removal efficiency of the mixture has also been tested in dynamic systems in the column study (Paper VI) and in the pilot plant study (Paper II). These studies show that a mixture of peat and carbon-containing ash is a good material for removal of pollutants from leachate. The used filter bed material in this study (Paper VII), taken from the full-scale treatment plant for industrial landfill leachate, at Halmstad, had been in use for 3 years when samples of the material were taken. A detailed description of the full-scale treatment plant can be found in [47].

The investigations were performed using batch tests with an equilibrium time of 24 hours. For characterization of the mixture before and after its usage, the standardized leaching test SS-EN 12457-4 [141] was used. In the sorption tests solutions with known concentrations of selected pollutants were used as liquid, and the measured concentrations in the eluate showed the sorption of the pollutants to the material and thus the removal of the pollutants from the tested solution.

Via physcio-chemical processes, as in a batch test at neutral pH, relevant information about removal efficiencies of metals and non-polar organic compounds such as PCBs can be achieved. However, concerning removal efficiencies of polar organic compounds with relatively high solubility in water, *e.g.* phenolic compounds, batch procedures with an equilibration time of 24 hours are not suitable. For polar substances, removal in authentic treatment systems is often due to microbial degradation processes. Thus, a microbiological environment is required [134] and establishment of microbiological communities in a filter takes time. Accordingly, it is advised to perform long-term experiments with dynamic column or pilot plant experiments in removal efficiency studies of such compounds (Paper II, Paper VI) and [135].

A very small fraction of all components in the unused mixture leached out, and the low leaching values confirm that a mixture of peat and carbon-containing ash is a good material for a filter-based treatment plant for leachate. Leaching values for the used mixture are generally low, except for DOC, compared to the limit values for non-hazardous waste, included in waste acceptance criteria in NFS 2004:10 [19]. Accordingly the material must be treated with respect to DOC before it can be deposited at a landfill for non-hazardous waste. However, the measured effective heating value was similar to values for other biological materials considered for incineration, and energy recovery from the used mixture in an incineration plant would be possible and would probably be a better alternative from an environmental point of view. The used leaching test method for solid waste seems to be a good tool for evaluation of pollutants leaching out from unused and used mixtures.

6. Conclusions and future perspectives

In this Thesis a methodology is described based on an analytical protocol for evaluation of hazards from landfill leachate and solid waste. The analytical protocol (Paper I) covers the determination of important pollutants and considers the toxic impact of environmental pollutants occurring in different wastewaters. It includes a strategy, in which organic marker substances are used for evaluation of the environmental impact of organic pollutants in wastewater. The strategy covers both polar and non-polar organic compounds, and relies on automated or semi-automated analytical systems. The parameters in the dynamic protocol should be changed depending on the type of investigated wastewater and the purpose of the investigation. The protocol can be applied to water, as well as to solid materials and air. In this thesis, the solid materials were investigated through leaching tests and the hazard of the material was evaluated in the resulting eluate liquid. The protocol has also been shown to be a good tool for evaluation of treatment procedures of landfill leachate.

To estimate the environmental threat of solid materials, leaching tests are central tools for material characterization. The performance of two EU-standardized leaching tests was investigated in Papers IV and V. The percolation test is utilized in columns, needed specific equipment and takes about 30 days to perform. The batch test takes 2 days and only ordinary laboratory equipment is needed. The results of the comparison of the eluates from the investigated materials show that the values from the batch test generally are higher with a few exceptions. For the purpose of checking whether the waste material complies with regulations, the batch test method, giving a conservative estimate in a shorter time than a column test, is to be preferred.

Experiences concerning the precision of leaching tests show that one generally has to expect large heterogeneity of the materials, which must be taken into consideration in a risk assessment. To be able to make a valid risk evaluation, it is important to perform a representative sampling including a sufficient number of replicates in each test. Furthermore, using demineralised water, as described in the EU tests, one will most probably underestimate pollutant values. In this Thesis it has been shown that the use of authentic leachate as leachant, leads to significantly increased concentrations of heavy metals in the eluate, compared to the use of demineralised water. However, uncertainty about the composition of the leachate could, for the sake of simplicity, favour a well-defined alternative such as demineralised water. On the other hand a choice of a synthetic leachant containing salts and/or ammonia would give a more conservative estimate of the risk of environmental hazards.

Long-term studies such as the pilot plant study (Paper II) and the column study (Paper VI) have shown, that the tested mixture of peat and carbon-containing ash is a good filter material for simultaneous removal of different types of marker pollutants from leachate. Also the short-term batch test (Paper VII) gave relevant information considering inorganic and non-polar organic marker compounds. For investigation of the removal of polar organic markers (*e.g.* phenolic compounds) a batch test is not sufficient and needs to be complemented with long-term studies, in which a microbial community has time to become established, giving the possibility for microbial degradation processes.

Determinations and chemical analyses of organic compounds are expensive, and it is difficult to evaluate the environmental risk from results of chemical analyses. Pollutants in leachate occur as complex mixtures and the risk of such mixtures cannot be adequately anticipated on the basis of effect and behaviour of individual components. Furthermore, it is the bioavailability fraction of the organic compound which is of greatest interest in a risk assessment. Therefore, risk assessment of polluted sites should include biological tests as well as information on the total amounts of pollutants. A biotest should yield some sort of ecological data, but this is seldom obtained with one single organism. For assessing the hazards from landfill leachate, the acute toxicity test with *Artemia salina* (Paper III) needs to be complemented with biological tests which utilize organisms representing different trophic levels in the ecosystem.

A further evaluation of the MARA system for acute toxicity investigations of landfill leachate would be interesting. This system is based on an array of eleven microorganisms (ten bacterial strains and one yeast) selected from different parts of the phylogenetic tree, in order to represent high genetic diversity. The system produces an array of responses from each microorganism to each compound that is tested, and a diverse pattern of toxicity values - “a toxic fingerprint” - from each tested chemical are generated. A further approach can be to test complex wastewater samples and compare the toxic fingerprints of unknown compounds to those on known compounds, of which extensive toxicity data are already available.

To evaluate any adverse effects that might evolve from a long-term exposure to a leachate, chronic toxicity tests are needed. A good future approach to assess endocrine disruption could be by monitoring the vitellogenin gene expression in male fishes, indirectly by spectrophotometric phosphate determination. This simple, inexpensive methodology could be a powerful tool in future investigations of landfill leachates.

There is no national regulation in Sweden that relates the characterization of leachate or the assessment of the threats from leachate on the environment or to human health. Local authorities stipulate outlet limit values according to determinations of volume and character of the leachate, given by the local landfill operator. Hopefully, in the future there will be national limits or guiding values for treated leachate to recipient to compare with.

One other lack in regulations is in the waste acceptance criteria. Neither the EU nor the Swedish waste acceptance criteria (WAC) gives any leaching limit values for organic parameters for non-hazardous and hazardous waste. The guidelines permit 5% TOC to landfills for non-hazardous waste, and this may include high concentrations of POPs. Modern landfills for non-hazardous and hazardous waste should be constructed with collecting and treatment systems for leachate. However, with no regulations concerning characterization of landfill leachate and no guidelines on leaching values of organic compounds in leaching tests, the spread of organic pollutants from solid waste into the environment cannot be controlled.

One attractive way to facilitate the evaluations of hazards from solid waste would of course be to reduce the waste volumes. This can be done by less consumption, re-use of products, re-cycling of materials, and by choosing environmentally friendly products when buying new things.

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8. Glossary

Aerobic	living or occurring only in the presence of oxygen
Aliquot	a portion of a total amount of a solution
Anaerobic	living or occurring only in the absence of oxygen
Analyte	substance to be measured
Bioaccumulation	occurs when an organism absorbs a toxic substance at a rate greater than that of excretion
Biodegradation	breakdown of organic compounds into carbon dioxide, water and minerals by the action of microorganisms
Bioindicator	an organism giving information about its habitat by its presence, absence or behaviour
Biomarker	a change in a biological response which can be related to an environmental chemical at the indvidual organism level, measured inside an organism or in its products, such as urine, blood, hair etc., indicating a deviation from the normal status
Congener	related chemicals, *e.g.* a derivative
Effluent	an outflow of water from a natural body of water, or from a man-made structure
Eluate	solution obtained by a laboratory leaching test
Eutrophication	an increase in chemical nutrients, typically compounds containing nitrogen or phosphorus, in an ecosystem.
Hazardous waste	waste with the potential to harm human health and/or the environment
Hydrophilic	soluble in water and in other polar solvents; a hydrophilic surface attratcs water and wets easily
Hydrophobic	insoluble in water; hydrophobic molecules tend to be non-polar and thus dissolve more effectively in non-polar solvents and associate with other hydrophobic molecules in prescens of water. Hydrophobic surfaces repel water and wet with difficulty

Inert waste	waste that does not undergo any significant physical, chemical or biological transformations. Inert waste will not dissolve, burn or otherwise physically or chemically react, biodegrade or adversely affect other matter with which it comes into contact in a way likely to give rise to environmental pollution or harm human health
Leachant	liquid that is brought into contact with the test portion in the leaching procedure
Leachate	the liquid that drains or 'leaches' from a landfill
Liquid to solid ratio	the ratio between the amount of liquid (L in litre) which had been in contact with a material, and the mass of a solid material (S in kg of dry matter)
Municipal waste	waste from households, as well as other waste which, because of its nature or composition, is similar to waste from household
Recipient	water, *e.g.* lake, river, and sea, that receives wastewater after treatment
Sewage	wastewater produced by human bodies, carried away from houses through special pipes to treatment plants

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