

A survey of toxic organics in Norwegian sewage sludge, compost and manure

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Sammendrag

Undersøkelser initiert av Statens forurensetingsstilsyn og Landbruksdepartementet, og gjennomført i samarbeid med bl.a. NORVAR, har gitt oppdatert informasjon om innholdet av 50 organiske miljøgifter i norsk avløpsslam, kompost og husdyrgjødsel. I perioden okt. 1996 - febr. 1997 ble det tatt 36 månedsblandprøver av ferdigbehandlet slam fra 8 større avløpsrenseanlegg, samt i alt 9 blandprøver av ferdig kompost fra 9 anlegg som komposterer våtorganisk avfall og 8 blandprøver av husdyrgjødsel (storfe og gris) fra 4 forskjellige gårder. Alle prøvene ble analysert for dioksiner/furaner, PCB, PAH, alkylfenoler, ftalater og LAS (anionisk detergent).

Analyseresultatene fra slamprøvene ble sammenlignet med data fra en tilsvarende slamundersøkelse i 1989, med tidligere svenske og danske undersøkelser samt med de retningsgivende verdier/grenseverdier som finnes i slamregelverkene i Sverige, Danmark og Tyskland.

Undersøkelsene konkluderer med at innholdet av organiske miljøgifter i norsk avløpsslam har gått ned i forhold til 1989-nivået, med unntak av PAH-innholdet som har holdt seg noenlunde konstant. Innholdet av nonylfenol og -etoksilater er imidlertid høyere enn i Sverige og Danmark og overskrider også verdiene i disse lands slamregelverk. Resultatene viser for øvrig store variasjoner fra måned til måned for hvert anlegg og indikerer klart at analyser av organiske miljøgifter på enkeltprøver av avløpsslam kan gi svært misvisende informasjon om nivået ved det enkelte anlegg.

Norske myndigheter har bl.a. på grunnlag av disse undersøkelsene, konkludert med at det ikke er grunnlag for å innføre grenseverdier for organiske miljøgifter i avløpsslam og kompost.

Abstract

A survey initiated by the Norwegian environmental authorities, has given

updated information about the content of toxic organics in Norwegian sewage sludge, compost and manure. 36 monthly composite samples of disinfected, stabilized and dewatered sludge from eight sewage treatment plants were analyzed for dioxins/furans, PCBs, PAHs, alkylphenols, phthalates and LAS (anionic surfactant). The same analytical programme was carried out for 9 samples of compost from source separated organic household waste and 8 samples of cattle and pig manure.

The data from this survey was compared to previous Norwegian, Swedish and Danish sewage sludge analyses and to Swedish, Danish and German standards. After an evaluation of all these aspects, the Norwegian authorities have decided not to include limit values for toxic organics in the existing regulations for sewage sludge and compost.

Introduction

In January 1995 the Norwegian health and environmental authorities jointly issued a new regulation for sewage sludge treatment and disposal (Ministry of Health and Social Welfare and Ministry of the Environment, 1995). This regulation was amended in September 1996, and the major amendment was a further reduction in the limit values for heavy metal content in sewage sludge to be applied on land areas.

The new regulation actively promotes those sludge management practices that provide for the beneficial use of sludge while maintaining or improving

environmental quality and protecting human health. One goal announced by the authorities is to recycle at least 75% of the total sewage sludge production for beneficial use on land areas by the year 2000. In achieving this, it is realised that only high quality sludge should be offered to agriculture and green areas; the two disposal routes acceptable for land application of sludge in Norway according to the new regulation. This means that the sludge should contain only very small amounts of toxic elements, and odour nuisance and health risks should be effectively controlled. The regulation, therefore, includes stringent standards for heavy metal content in both sludge and soil. Furthermore there is a general requirement for stabilisation and disinfection of all sewage sludges before land application. In addition to emphasising sludge quality the regulation also sets limits for land application rates, and the type of crops to be grown on sludge applied areas, and forbids growing of potatoes, vegetables, and fruits for 3 years after sludge application. Further details are given in Paulsrud and Nedland, 1997.

In September 1996 the Ministry of Agriculture launched a revised version of the regulation for the use of organic waste products in agriculture and for land reclamation, parks and lawns. (Ministry of Agriculture, 1996). This regulation covers a wide range of waste products, including compost from source separated organic household waste, growth media containing sewage sludge (max 30% sewage sludge) and

organic fertilizers with sewage sludge origin. The sewage sludge and organic waste regulations are harmonised regarding criteria for heavy metal content, but there are no limit values or guidelines for the content of toxic organics.

As a result of media focus during 1995-1996, farmers and environmental organisations became increasingly concerned about potential oestrogenic compounds in sewage sludge. This situation forced the authorities to present updated information about toxic organics in waste products spread on agricultural land, and a survey was initiated.

The main objectives of the survey were to:

- present representative data for the content of certain toxic organics in sewage sludge and compost, and to compare these with the corresponding data for animal manure
- compare the data with previous Norwegian analyses of sewage sludge and recent data from Denmark and Sweden, and with the German, Danish and Swedish regulations on toxic organics (organic xenobiotics) in sewage sludge and compost
- evaluate the need for including limits for toxic organics in the Norwegian regulations governing sewage sludge and compost.

Materials and methods

Sewage treatment plants, compost plants and farms included in the survey

Tables 1, 2 and 3 present some basic information about the plants and farms included in the survey. The sewage treatment plants were randomly selected among those plants treating both municipal and industrial wastewater and with experience from sludge recycling and the reduction of toxic elements in sewage sludge. The criteria for the selection of composting plants were the use of source separated household waste as the major organic waste to be composted, and that sewage sludge is not handled. The farms for manure sampling were randomly selected, except that one of them should operate without utilising mineral fertilizers and pesticides ("ecological farming").

Selection of parameters to be analysed. Analytical procedures

The following criteria were used to determine the range of compounds to be analysed in the study:

- Listed as a high priority contaminant by the health and environmental authorities
- Detected in fairly high concentrations in the previous (1989) sewage sludge study (Vigerust, 1989)
- Included in other countries sewage sludge or compost regulations or

Table 1. Sewage Treatment Plants Included in the Survey

Plant no.	Plant size (actual load, pe)	Sewage treatment process	Coagulant for P-removal	Sludge treatment process *)
1	250,000	Primary - chemical	Fe-chloride	Anaerobic digestion
2	75,000	Primary-chemical	Fe-chloride	Pre-pasteurisation + anaerobic digestion
3	65,000	Biological - chemical	Alum	Thermal hydrolysis + anaerobic digestion + thermal drying
4	81,000	Primary - chemical	Fe-chloride	Pre-pasteurisation + anaerobic digestion
5	75,000	Primary - chemical	Al-chloride (prepol.)	Indoor windrow composting with bark
6	67,500	Primary - chemical	Fe - chloride	Lime treatment
7	480,000	Biological -chemical	Al-chloride (prepol.)	Anaerobic digestion + lime conditioning
8	40,000	Primary-chemical	Fe-chloride	Thermophilic aerobic pretreatment + anaerobic digestion

*) All plants included in the survey employ both gravity thickening and mechanical dewatering in addition to the processes listed in the table.

Table 2. Composting Plants Included in the Survey

Plant no.	Type of composting plant	Organic waste to be composted	Bulking agents
1	In vessel (reactor)	Source separated household waste	Bark + garden waste
2	In vessel (reactor)	Source separated household waste	Wood chips + garden waste
3	Windrow	Source separated household waste + some horse manure	Mainly garden waste + some bark and wood chips
4	Windrow	Source separated household waste included nappies	Garden waste
5	Windrow	Source separated household waste included nappies	Garden waste
6	Windrow	Source separated household waste included nappies	Garden waste
7	Windrow	Kitchen waste from hotels and restaurant	Garden waste
8	Windrow	Source separated household waste	Garden waste
9	Windrow	Source separated household waste +some poultry manure	Garden waste + wood chips

Table 3. Origin of Manure Included in the Survey

Farm no..	Way of operation	Type of manure
1	Ecological	Cattle
2	Conventional	Cattle
3	Conventional	Pig
4	Conventional	Cattle

guidelines (Danish Ministry of Environment and Energy, 1996; Der Bundesminister für Umwelt, Naturschutz und Reaktorsicherheit, 1992; National Swedish Environmental Protection Board, 1994)

- Certain limits for the total cost of laboratory analyses

Six main groups of organic compounds were selected based on the given criteria:

dioxins/furans, PCDD/PCDF (17 compounds), PCBs (7 congeners), PAHs (16 compounds), alkylphenols (nonylphenol/-ethoxylates + 2-methylphenol + 3/4-methylphenol), phthalates (DEHP + DBP) and linear alkylbenzene sulfonates (LAS).

Eight Scandinavian laboratories were invited to tender for provision of the laboratory services, and the successful laboratories were the Norwegian Institute for Air Research and the Swedish KM Lab. Table 4 summarises some key information about the analyses performed.

Sampling procedures

All sampling equipment (gloves,

spoons, containers) were selected and prepared by the laboratories to avoid contamination of the samples, and detailed instructions were worked out on how to perform the sampling of the different materials at each plant/farm.

The sewage sludge samples were taken as monthly composite samples from each plant in the period Oct. 1, 1996- Febr. 28, 1997. These samples were composed of approx. 30 daily composite samples representing each days production of treated sludge ready for land application. All samples were kept frozen until they were analysed.

Only one compost sample from each composting plant was taken during the period Dec. 96 - Jan. 97. These samples were made up of about 10-20 grab samples from different places in the heaps of compost ready for delivery. All samples were transported directly to the laboratories without any conservation (freezing).

Samples of animal manure were taken as grab samples from the slurry tanks, and each farm was sampled twice within a period of about one month during Dec. 1996. These samples were frozen prior to analysis.

Table 4. Analytical methods, detection limits and uncertainties

Parameter	Method	Detection limit	Uncertainty
PCDD/PCDF I	GC-MS, NILU - O - 1 ¹⁾	0,1 ng/kg dw	± 5%
PCBs	GC-MS, SNV 3829 (mod.) ²⁾	0,001 mg/kg dw ³⁾	± 20% ⁴⁾
PAHs	GC-FID/MS, SNV 3829 (mod.) ²⁾	0,1 mg/kg dw ³⁾	± 30% ⁴⁾
Alkylphenols	GC-ECD/MS, SNV 3829 (mod.) ²⁾	1 mg/kg dw	± 34%
Phthalates	GC-MS/SNV 3829 (mod.) ²⁾	1 mg/kg dw	± approx. 30% ⁵⁾
LAS	Standard Methods 555C (mod.)	1 mg/kg dw	± approx. 30% ⁵⁾

- 1) The method refers to an internal, accredited procedure of the Norwegian Institute for Air Research
- 2) Refer to publication no. 3829 from the National Swedish Environmental Protection Board (SNV) (1990)
- 3) Apply to each of the PCB congeners and the sum of 6 PAH compounds
- 4) Apply to the sum of 7 PCB congeners and the sum of 6 PAH compounds
- 5) These two analyses were not accredited.

Results and discussion

Table 5 (sewage sludge) and table 6 (compost and manure) summarise the results of the study. Table 7 presents a comparison between this study and previous Scandinavian investigations of toxic organics in sewage sludge. Figure 1 compares the levels of toxic organics in sewage sludge, compost and manure measured in this study.

Dioxins/furans (PCDD/PCDF)

The concentration of PCDD/PCDF in the sewage sludge was in general very low and showed only small monthly variations. Only one plant (# 7) had real peak values, but still they were below the maximum values in the German standard. Some of the compost samples

had PCDD/PCDF-content at the same level as the sludge samples, while the manure concentrations were just above the detection limit.

PCBs

The PCB content of the sludge samples was low and, in general, lower than that found in previous studies in Scandinavia. However, there were great variations between monthly samples from each plant, and in fact greater than between samples from the different plants. All values were far below the German and Swedish standards for PCB. For compost and manure there were the same situation as for dioxins/furans.

Table 6 Toxic Organics in Norwegian Compost and Manure 1996 - 97

Composting plant no.	PCDD/PCDF ng I-TE/kg dw	PCB		PAH		3/4 methylphenol mg/kg dw	Nonylphenol/-ethoxylates mg/kg dw	Phthalates		LAS mg/kg dw
		Sum 7 PCBs mg/kg dw	Sum 6 PAHs mg/kg dw	Sum 10 PAHs mg/kg dw	Sum 16 PAHs mg/kg dw			DBP mg/kg dw	DEHP mg/kg dw	
1	6,15	0,0283	1,59	2,39	2,59	n.d.	n.d.	7,9	<1	22
2	3,72	0,0207	0,13	0,13	0,55	n.d.	n.d.	2,9	13	75
3	5,73	0,0198	2,37	3,26	3,77	n.d.	n.d.	4,8	<1	396
4	3,17	0,0187	0,76	0,97	1,24	n.d.	n.d.	2,9	1,4	24
5	11,90	0,0777	0,54	0,54	0,92	n.d.	n.d.	5,4	4	67
6	6,92	0,0185	1,00	1,49	1,92	n.d.	n.d.	1,6	26	41
7	0,49	0,0027	n.d.	n.d.	0,28	n.d.	n.d.	2,8	<1	78
8	0,83	0,0145	0,62	0,62	1,01	n.d.	n.d.	<1	9	9
9	0,76	0,0182	n.d.	n.d.	n.d.	22	n.d.	<1	12	34
Min.	0,49	0,0027	n.d.	n.d.	n.d.	n.d.	n.d.	<1	<1	9
Median	3,72	0,0187	0,62	0,62	1,01	n.d.	n.d.	2,9	1,4	41
Mean	4,41	0,0241	0,78	1,04	1,36	2,0	n.d.	3,1	6,3	83
Max.	11,90	0,0777	2,37	3,26	3,77	22,0	n.d.	7,9	26	396
Farm no. *)										
1A	0,56	0,0044	n.d.	n.d.	n.d.	110	n.d.	2,0	<1	150
1B	0,26	0,0026	n.d.	n.d.	n.d.	195	n.d.	2,5	21	65
2A	1,02	n.d.	n.d.	0,54	0,54	99	n.d.	<1	3,7	100
2B	0,27	0,0013	n.d.	n.d.	n.d.	28	n.d.	1,9	26	58
3A	0,42	0,0013	n.d.	n.d.	n.d.	194	n.d.	<1	<1	27
3B	0,24	0,0014	n.d.	n.d.	n.d.	280	n.d.	<1	3,3	14
4A	0,24	0,0041	n.d.	n.d.	0,46	245	n.d.	<1	<1	185
4B	0,32	0,0028	n.d.	n.d.	n.d.	n.d.	n.d.	3,2	<1	77
Min.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	<1	<1	14
Median	n.d.	0,0020	n.d.	n.d.	n.d.	152,0	n.d.	1,0	1,7	71
Mean	0,42	0,0022	n.d.	0,07	0,13	144,0	n.d.	1,2	6,8	85
Max.	1,02	0,0044	n.d.	0,54	0,54	280,0	n.d.	3,2	26	185
Guidelines and regulations for toxic organics in sewage sludge										
Swedish guideline (1994)		0,4 ^a	3				100 ^{aaa}			
Danish regulation 01/07/97				6			50		100	2 600
Danish regulation 01/07/00				3			10		50	1 300
German regulation (1992)	100	0,2 ^{aa}								

^a Annual average

^{aa} Valid for each of 6 PCB congeners

^{aaa} The value was reduced to 50 mg/kg dw from 01/01/97

*) A and B represent two different samples

n.d. = not detected (below detection limit)

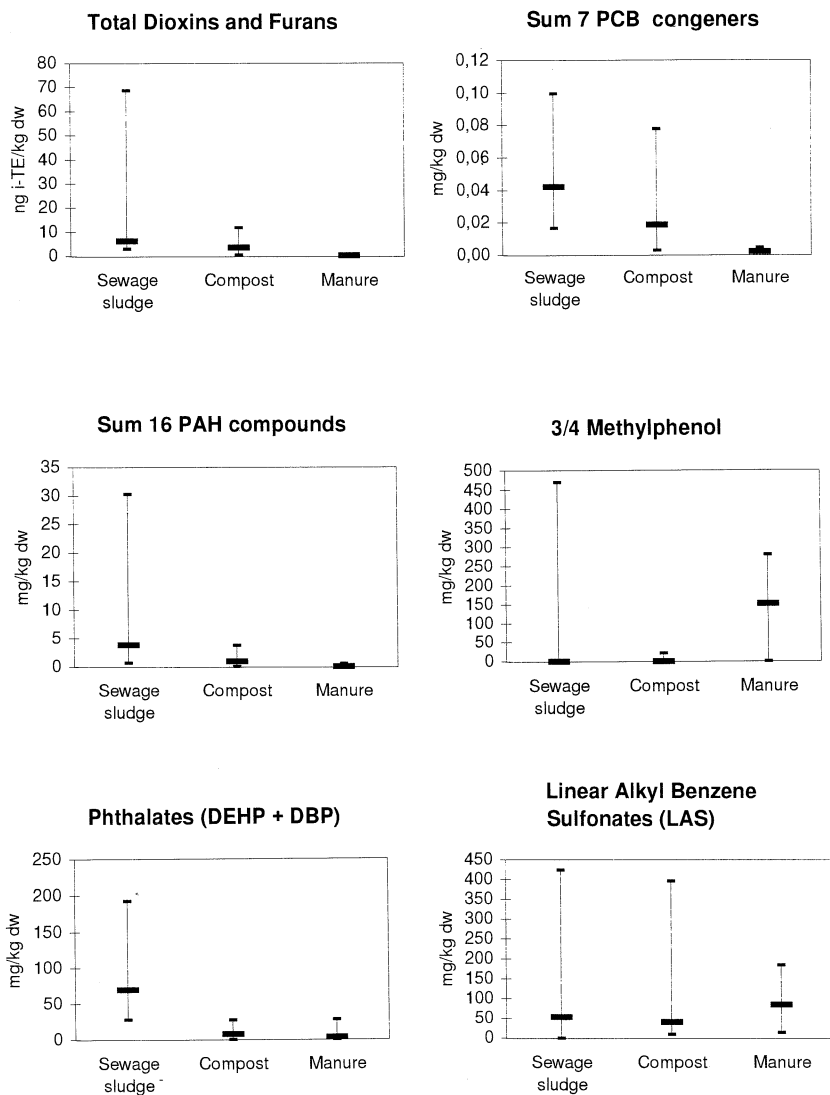


Figure 1. Comparison of toxic organics in Norwegian sewage sludge, compost and manure (minimum, median and maximum values).

Table 7. Comparison of Scandinavian Investigations of Toxic Organics in Sewage Sludge

Parameter	Investigations	No. of samples	Range	Median	References
Dioxins/furans (ng i-TE/kg dw)	This study	36	3,0-68,8	6,26	National Swedish Environmental Protection Board, 1992 Tørsløv et. al., 1997
	Swedish (1989-91)	14	5,7-115	20,5	
	Danish (1993-94)	9	10,3-34,2	–	
PCBs (Sum of 7 PCB congeners) (mg/kg dw)	This study	36	0,0168-0,0996	0,0422	National Swedish Environmental Protection Board, 1995 National Swedish Environmental Protection Board, 1992 Tørsløv et. al., 1997 Tørsløv et. al., 1997
	Swedish (1993)	23	0,0006-0,232	0,113	
	Swedish (1989-91)	27	0,080-7	–	
	Danish (1995)	20	<0,027-0,186	–	
	Danish (1993-94)	9	<0,030-0,140	–	
PAHs (Sum of 16 PAH compounds) (mg/kg dw)	This study	36	0,7-30	3,9	Vigerust, 1989 National Swedish Environmental Protection Board, 1995 National Swedish Environmental Protection Board, 1992 Tørsløv et. al., 1997 Tørsløv et. al., 1997
	Norwegian (1989)	19	<1,0-24 ¹⁾	<1,0 ¹⁾	
	Swedish (1993)	23	<0,3-4,9 ²⁾	2,0 ²⁾	
	Swedish (1989-91)	27	24- 199 ²⁾	–	
	Danish (1995)	20	<0,01-8,5 ³⁾	–	
	Danish (1993-94)	9	0,42-2,4 ³⁾	–	

Nonylphenol (+ ethoxylates) (mg/kg dw)	This study	36	22-650	136	Vigerust, 1989 National Swedish Environmental Protection Board, 1995 National Swedish Environmental Protection Board, 1992 Tørsløv et. al., 1997 Tørsløv et. al., 1997
	Norwegian (1989)	19	25-2298	189	
	Swedish (1993)	23	23-171	82	
	Swedish (1989-91)	27	44-7214	825	
	Danish (1995)	20	0,3-67	8	
Phthalate, DEHP (mg/kg dw)	Danish (1993-94)	9	55-537	—	Vigerust, 1989 National Swedish Environmental Protection Board, 1992 Tørsløv et. al., 1997 Tørsløv et. al., 1997
	This study	36	<1-140	58	
	Norwegian (1989)	19	27-1115	83	
	Swedish (1989-91)	27	25-661	170	
	Danish (1995)	20	3,9-170	24,5	
LAS (mg/kg dw)	Danish (1993-94)	9	17-120	38	Tørsløv et. al., 1997 Tørsløv et. al., 1997 Tørsløv et. al., 1997 Tørsløv et. al., 1997 Tørsløv et. al., 1997
	This study	36	<1-424	54	
	Danish (1995)	20	11-16100	530	
	Danish (1993-94)	6	200 - 4640	455	
1) Sum of 10 PAH compounds					
2) Sum of 6 PAH compounds					
3) Sum of 18 PAH compounds					

PAHs

The PAH content was low in most sewage sludge samples and well below the Swedish and Danish standards (as of 1997). However one plant (# 3) exhibited high values in 4 of 5 samples, and one sample from plant 6 was over the standards. Again large monthly variations were witnessed for most plants, suggesting that one single sample is not sufficient to state the level of toxic organics in sewage sludge. The PAH concentrations measured in this study are almost at the same level as in the previous Norwegian investigation (Vigerust, 1989), but above the more recent values reported in Sweden and Denmark (National Swedish Environmental Protection Board, 1995; Tørsløv et al., 1997).

Some of the compost samples had PAH contents as high as the best sludge samples, but in general they were much lower, and the manure samples didn't show PAHs above the detection limits except two samples.

Alkylphenols

Nonylphenol (+ ethoxylates) were found in high concentrations in sludge samples from all the sewage treatments plants in the survey, and all the plants would have been classified as non-compliant with the Swedish and Danish standards. There has only been a minor decrease in nonylphenol concentration in Norwegian sludges since 1989, while Sweden and Denmark have experienced a much greater reduction during the nineties. This is mainly due to their exertion of pressure on the in-

dustries to phase out this compounds from their products (i.e. detergents, paints). Similar experiences have been reported from Switzerland (Giger, 1997). No nonylphenol (+ ethoxylates) has been detected in any of the compost and manure samples in this study.

3/4 methylphenol (m-/p-cresol) were detected in fairly high concentrations in manure from all farms, while only one sewage treatment plant (# 3) gave similar (or higher) concentrations in the sludge. These compounds are intermediates in the decomposition of amino acids in man and animals, and will rapidly decompose further under aerobic conditions. The high content in sludge from plant no. 3 may be explained by the thermal hydrolysis process employed at this plant.

Phthalates

DEHP was detected in almost all sewage sludge samples, and three of the plants revealed concentrations above the Danish 1997-standard. DBP was detected less frequently and also at lower concentrations than DEHP. There has been a significant reduction in DEHP content of Norwegian sludges since 1989, but the values are still higher than in the Danish investigations. Both DEHP and DBP were also found in compost and manure, but at lower levels than in sewage sludge.

LAS

The LAS content of sewage sludges in this study was very variable, but in general the values are far below the Da-

nish standard and the concentrations reported in the Danish investigations (Tørsløv et al., 1997). This can partly be explained by the different analytical methods used, but also to the fact that most Norwegian households use eco-labeled detergents, indicating that they do not contain LAS. These compounds were also found in compost and manure at similar levels to the sewage sludge.

Conclusions

1. Analyses of five monthly composite samples of sewage sludge from seven wastewater treatment plants, revealed that the monthly variations in toxic organic content are substantial for most of the parameters analysed, and that the variations within each plant can be even greater than the variations between different plants. This clearly indicates that toxic organic analyses of single daily composite samples of sewage sludge can provide very misleading information about the level of organic pollutants.
2. There has been a significant reduction of nonylphenol (+ ethoxylates) and phthalates (DEHP) in Norwegian sewage sludges between 1989 and 1997. However, the PAH content of the sludges has remained fairly constant during this period.
3. In comparing the content of toxic organics in Norwegian sludges with the sewage sludge regulations in Denmark and Germany and the guideline in Sweden, we can conclude that all plants in the survey would comply with the given limit values for dioxins/furans, PCBs and LAS. Two plants would not meet the standards regarding PAH-content and three plants had greater DEHP-contents than those allowed in the Danish regulation. None of the plants comply with the nonylphenol standards in Denmark and Sweden.
4. The content of toxic organics in nine Norwegian compost samples from source separated organic household wastes was at the same level as in the Norwegian sewage sludge with the best quality, except for nonylphenol which were below the detection limit in all compost samples.
5. Eight samples of pig and cattle manure revealed very low concentrations of toxic organics, except for 3/4 methylphenol and LAS which were at the same level as in Norwegian sewage sludge.
6. Based upon the results of this survey, the Norwegian authorities have decided not to include limit values for toxic organics in the existing regulations for sewage sludge and compost. In order to reduce the high sewage sludge content of nonylphenol (+ ethoxylates), a phasing out of these components in domestic and

industrial products has been intensified, and shall be completed within the year 2000.

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