

BEHAVIOUR OF EDC IN WWTP AND MODEL SYSTEM – PREDICTIVE MODEL ON THE BEHAVIOR AND FATE OF EDCs IN WWTPs

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I. OBJECTIVES FOR THE REPORTING PERIOD

Transport, behaviour and fate of major estrogenic endocrine disrupting compounds during wastewater treatment should be studied in laboratory experiments as well as in full-scale wastewater treatment plants. Both the water and the solid phase should always be analysed for the target compounds.

17 β -estradiol, 17 α -ethinylestradiol, bisphenol A, and nonylphenolmono- and -diethoxylate, mestranol, estrone, estriol, 4-*tert*-octylphenol, and 4-nonylphenol were chosen as target compounds.

Laboratory experiments should be carried out in order to study the degradation of 17 β -estradiol, 17 α -ethinylestradiol, bisphenol A, and nonylphenolmono- and -diethoxylate. Since oxidation is the main mechanism of the microbiological degradation of these phenolic compounds, simultaneous aerobic sludge treatment was applied for the realisation of the experiments. A descriptive technical model should be found that shows the fate of each of these compounds during these experiments.

Field samplings had to be carried out at full-scale plants treating wastewater by simultaneous aerobic sludge stabilisation.

Sampling strategies should be confirmed or, if necessary, improved.

Subcontractors should carry out YEAST assays (GEW) and on-line monitoring of EDC (SCREEN TEC).

II. OUTCOME AND DELIVERABLES

Behaviour and fate of EDCs in WWTPs

- Elimination kinetics of bisphenol A (BPA), 17 β -estradiol (E2), estrone (E1), 17 α -thinylestradiol (EE2) and nonylphenolmono- and -diethoxylate (**NP₁EO**, **NP₂EO**, **NP_{1,2}EO**) from synthetic wastewater.
- Mass balances were **calculated** to **model** the partitioning of the substances between sewage sludge and wastewater in the systems.
- Descriptive model: descriptive models could be obtained describing the behaviour (elimination, formation and partitioning between fluid and soild phase) of bisphenol A, 17 β -estradiol and estrone in WWTPs with denitrification and aeration.
- New analytical **techniques** for the determination of endocrine disruptors (EDCs) in wastewater and sewage sludge were developed, optimized and validated.

III. MAIN RESULTS

1. Behaviour of EDC under terms of controlled lab scale plants and modelling

Analytics

Liquid samples are centrifuged, conserved and run through a solid phase extraction (SPE) cartridge. Solid samples are freeze-dried and subsequently treated by Soxhlet extraction and size exclusion chromatography (SEC). Afterwards an additional clean-up by means of silica gel as well as derivatization (silylation) and determination by gas chromatography/mass spectrometry follow, respectively. Solvents of HPLC grade are used for SPE and SEC column conditioning and for sample preparation. Extracts were analyzed for natural and synthetic steroids as well as xenoestrogens by gas chromatography / mass spectrometry (GC HP 6890, MSD HP 5973, phenyl-methyl-siloxane column HP-5MS, 5 %, 30 m x 250 μm x 0.25 μm nominal, Hewlett Packard, Boeblingen, Germany, respectively). The target EDC compounds determined are 4-tert-octylphenol (OP), 4-nonylphenol (NP), bisphenol A (BPA), estrone (E1), 17 β -estradiol (E2), estriol (E3), mestranol (M), and 17 α -ethinylestradiol (EE2). For details see ANNEX I.

1.1. Lab Scale Wastewater Treatment Plant Units and Realisation of Experiments

The laboratory wastewater treatment plant (WWTP) type used (behrotest[®] KLD4, Behr, Duesseldorf, Germany) and the realization of the experiments basically met the regulations of OECD guideline 303A (OECD, 2001) and the German guideline DIN EN ISO 11733 (DIN, 1998). The minor changes made in comparison to these guidelines are described below or within the text of the respective chapter 1.2 to 1.5.

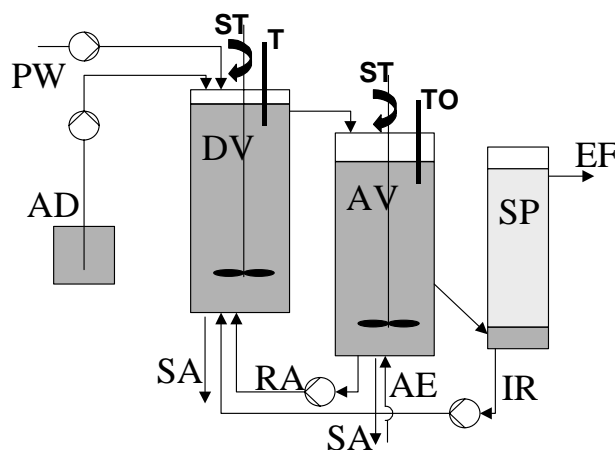


Figure 1: Experimental set-up and realization schematic diagram; AD - analyte & diet influent solution, AE - aeration, AV - aeration vessel, DV - denitrification vessel, EF - effluent, IR - recirculation of excess sludge / intern reflux, PW - potable water influent, RA - recirculation of activated sludge / denitrification reflux, SA - sampling of sludge suspension, SP - separator vessel, ST - stirrer, T - temperature probe, TO - temperature and oxygen probe

Each WWTP consists of the diet reservoir, an anoxic and an aerobic treatment vessel ($V = 3$ l, respectively), the separator vessel ($V = 2.0$ l), oxygen and temperature measuring devices, stirrers, and an aeration system (see figure 1). Potable water (PW) and the diet / analyte solution

(AD) were supplied separately to the denitrification vessel (DV) resulting in a debit volume flow of 0.5 l/h. Thus, the debit hydraulic detention time of the sludge within the two activated treatment stages amounted to 12 h. The sludge suspension subsequently flowed through the aeration vessel (AV) and the separator (SP). The denitrification as well as the aeration vessel were continuously stirred. Both activated sludge and excess sludge were in part led in a closed loop leading to an overall sludge return ratio of 1.6. The aeration system automatically regulated the oxygen concentration in the aeration vessel between 2 and 4 mg O₂/l.

Table 1: OECD and DIN EN ISO criteria for success of degradation experiments by means of simultaneous aerobic sludge treatment (DIN, 1998, OECD, 2001)

parameter	target value
COD / DOC elimination rates	> 80 %
analyte degradation rate	> 90 %
nitrite-N concentration in effluent	< 0.61 mg/l
nitrate-N concentration in effluent	< 10 mg/l
ammonia-N concentration in effluent	< 0.78 mg/l
dry matter concentration in aeration vessel	2.5 g/l
pH value denitrification vessel	7.5 + 0.5
O ₂ concentration in aeration vessel	> 2 mg/l
temperature	18 – 25°C

In order to control the general degradation efficiency of the biocoenosis as demanded by the guidelines mentioned above, Chemical Oxygen Demand (COD), Dissolved Organic Carbon (DOC), nitrate-N, nitrite-N, and ammonia-N concentration, pH and conductivity of the influents, the effluents, and the supernatants of the denitrification and the aeration vessels have been determined at the beginning of every week, respectively. Table 1 shows the target values of the corollary parameters defined by the OECD and DIN EN ISO guidelines.

Samples have been taken of the mixed influent solution as it dropped into the denitrification vessels, out of the denitrification and aeration vessels, respectively, and of the effluents. The debit influent concentrations of the target compounds were set similar to environmentally occurring concentrations in raw municipal wastewater (0.1 to 10 µg/l, depending on target compound). Due to these low concentrations, weekly composite samples of 1 l were necessary to take for triplicate determinations. Therefore, from Monday through Friday 0.2 l sludge suspension, influent, and effluent have been drawn, respectively. The debit sludge age amounted to 20 to 21 d as frequently applied in full-scale WWTPs.

The units have not been cross-linked except from the fact that they were inoculated by parts of the same municipal sewage sludge and that the diet influent solutions were supplied from the same stock.

1.2. 17 β -estradiol degradation experiments

Set-up and realisation

The 17 β -estradiol degradation experiments were carried out from March to June 2000. The debit E2 influent concentration amounted to 1 $\mu\text{g/l}$. In week 9 the aeration unit of WWTP 1 was out of order for a time of 1 to 2 days. That caused anaerobization of the aeration vessel. No samples were drawn out of the denitrification vessels in week 11 and 12, respectively. In week 11 the sludge colour started to change via grey to black. This change of colour is typical for anaerobization of the sludge suspension **and the formation** of H₂S. Since this problem could not be solved, sludge suspension samples have not been taken or analyzed with the beginning of week 11. The volume fluxes were regulated before the first sampling for the determination of E2. The mass balance for WWTP 2, week 8, then was calculated by means of the debit volume fluxes (PW influent 446 ml/h, AD solution influent 54 ml/h, RA reflux of activated sludge for denitrification 1000 ml/h, IR reflux of excess sludge 550 ml/h).

Corollary parameters

The values of the corollary parameters met the target values in the most cases. The effluent nitrate-N concentrations exceeded the target values by about 50 % (see table 21 to table 31). The inorganic nitrogen data are in part inconsistent.

Concentrations

Probably because of the varying performance of the peristaltic pump, the influent E2 concentrations in several weeks distinctly differed from the debit value (see tables 32, 35). For the most weeks, the data are inconsistent. The sludge dry matter E2 and E1 concentrations were not detectable (n.d.) or not quantifiable (n.q.) in 2 third of the cases. That is not meaningful and stands in contradiction to the considerably high amounts in the cases of the other third of the samples (see tables 32, 33, 35, 36). Except of one case per WWTP, all water phase E2 concentration were $\leq 0.07 \mu\text{g/l}$. In the sludge DM, the E2 concentrations amounted to $\leq 12 \mu\text{g/kg DM}$.

E1 could be determined in all influent samples. Thus, E2 degradation in the diet reservoir and / or in the inlet tubes must be stated (see tables 33, 36). In the denitrification vessels, the liquid phase E1 concentrations amounted to $\leq 0.31 \mu\text{g/l}$, the solid phase E1 concentrations to $\leq 19.4 \mu\text{g/kg DM}$. In the aeration vessels, the liquid phase E1 concentrations were one order of magnitude lower than in the denitrification vessels. The AV sludge E1 concentrations amounted to $< 17.0 \mu\text{g/kg DM}$. Obviously, E2 was degraded to E1 especially in the denitrification vessels. Afterwards, E1 was degraded in the aeration vessels. The effluent E1 concentrations amounted to $\leq 0.021 \mu\text{g/l}$.

E3 was n.d. in the water phases except in one case (see tables 34, 37). Up to $\leq 22.4 \mu\text{g/kg DM}$ were detected in the aeration vessel sludge samples. Thus, degradation of E1 to E3 can be stated. The four AV sludge E3 concentrations determined in the range of 15.9 to 22.4 $\mu\text{g/kg DM}$ show that E1 is degraded to E3. The little number of valid E3 data does not allow to make further estimations.

Mass balances

In order to be able to calculate mass balances for WWTP 2, week 8, the result n.d. in the vial was replaced by 1.5xMDL, n.d. in the sample by LOD minus blank, and n.q. in the sample by LOQ minus blank. Thus, the figures always represent the highest value possible. No mass balance could be calculated for E3.

The E2 mass balance (figure 2) shows that 9.3 $\mu\text{g/d}$ were degraded already in the denitrification vessel and additional 1.8 $\mu\text{g/d}$ in the aeration vessel (see also table 39). In relation to the AD influent, these amounts represent 83 and 16 %, respectively. In relation to the respective overall

vessel influent it means that about 70 % of the E2 was degraded in the DV, and about 40 % in the AV (see table 2).

Adsorption of E2 to the sludge appeared in the denitrification vessel. The value of 0.67 $\mu\text{g/d}$ represents the lower limit of adsorption because it cannot be estimated if E2 was degraded only in the water phase or also when adsorbed to the activated sludge.

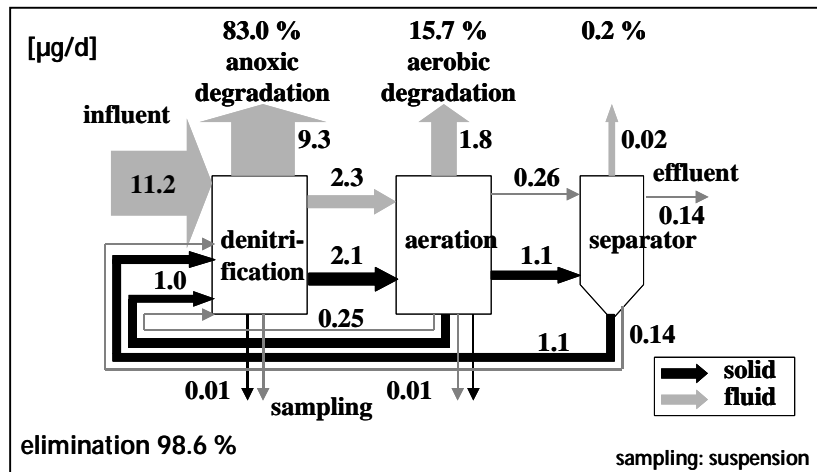


Figure 2: 17-estradiol mass balance of WWTP 2, week 8; elimination rate in relation to system AD+PW influent; all values [$\mu\text{g/d}$] except otherwise stated

Table 2: Elimination rates of the three vessels DV, AV, and SP, in relation to the respective total vessel influent; E2 degradation experiment, WWTP 2, week 8; all values [%], based on molar concentrations (see tables 38, 39); total system elimination rate calculated from difference between influent and effluent mass flux, respectively

[%]	E2	E1	E2+E1
denitrification vessel	67.7	-257.8	-6.6
aeration vessel	39.7	71.2	63.8
separator	1.2	12.1	-6.9
total system	98.6	59.8	97.9

The **low elimination rate** in the SP **possibly** was calculated due to the underestimation of the dry matter content of the **activated** sludge led back to the denitrification vessel (intern reflux, IR). **Another possibility is the overestimation of the AV sludge E2 concentration:** The separation of solids from the water is the original use of the separator and thus **causes a certain detention time of the sludge**. Since samples could not be taken neither from the separator nor from the IR, the masse balances have been calculated setting the target compound concentration of IR sludge equal to those of the AV sludge. Since 80 % of the IR mass flux is carried by the sludge an underestimation of the dry matter content **as well as the overestimation of the sludge E2 concentration could** cause a reasonable error. Sampling of the IR sludge was prevented **due to** the low system volume of about 8 l. The elimination of E2 in the SP is **one of the error values of this mass balance representing dry matter loss with the effluent, data variability, the inexact estimation of the dry matter concentration of the excess sludge, etc.** The value of 0.6 µg/d can be estimated as very low.

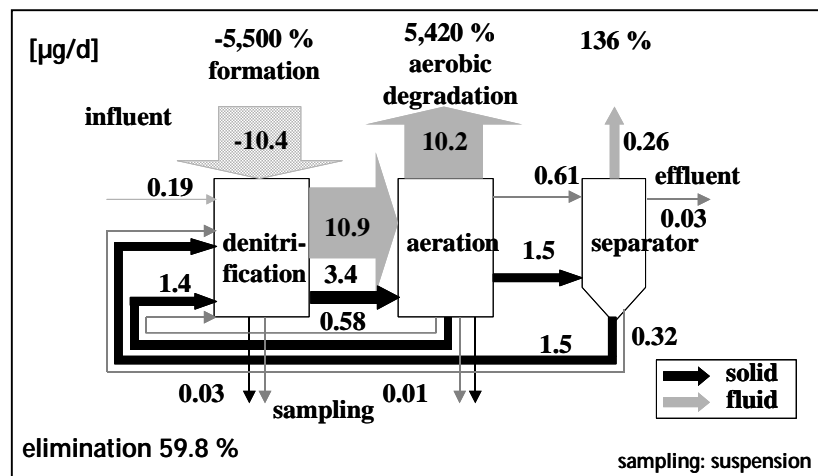


Figure 3: Estrone mass balance of WWTP 2, week 8; elimination rates in relation to system AD+PW influent; all values [µg/d] except otherwise stated

Very high amounts of E1 are formed in the denitrification vessel and afterwards degraded in the aeration vessel (see figure 3, table 39). Since only very low (and undesired) amounts of E1 are in the system AD influent the formation or elimination rates calculated in relation to the AD influent are very high and misleading. The mass of at least 10.4 µg E1 was formed per day in the denitrification vessel and at least 10.5 µg E1 were degraded per day in the aeration vessel. **Note that** the overall E1 elimination rate of about 60 % was **always** calculated **using the difference between** the total system influent and effluent data. It gives no information about formation and degradation of E1 within the certain vessels. Without the undesired E1 formation in the AD reservoir and inlet tubes, this rate even should be negative. In relation to the respective total vessel influent, formation of about 260 % appeared in the DV, but 70 % elimination in the AV.

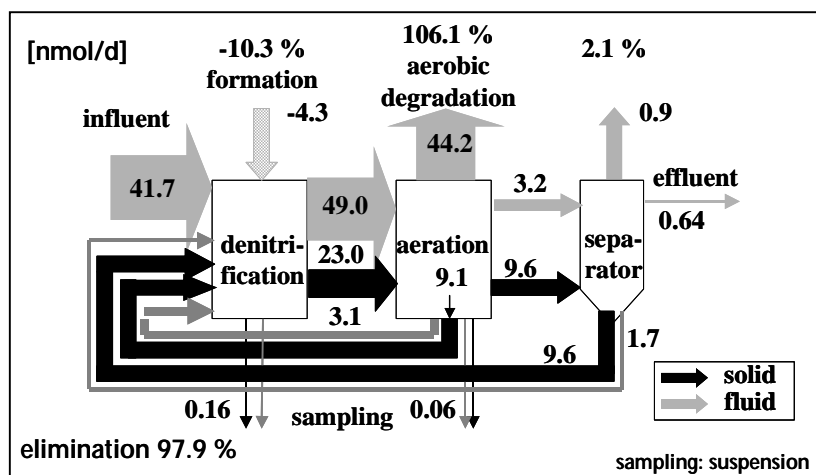


Figure 4: Summarised (17β-estradiol + estrone) mass balance of WWTP 2, week 8; elimination rates in relation to system AD+PW influent; all values [nmol/d] except otherwise stated

The summarized mass balance of E2+E1 calculated on **molar** basis is shown in figure 4 (see also table 38). The overall elimination rate amounted to 98 %. In the denitrification vessel, only E2 was degraded to E1 but not E1 to E3. The degradation of E1 to E3 was observed only under aerobic conditions in the aeration vessel. An increase of the sum E2+E1 in the denitrification vessel can be excluded. This fictitious formation of 10.3 % **possibly was caused by a varying and in this case underestimated IR volume flux. The volume fluxes have been assumed to be constant or linear over time and as high as the mean volume flux of two determinations, resp.** The **AV** elimination rate in relation to the respective total vessel influent amounted to 64 %.

1.3. 17α-Ethinylestradiol degradation experiments

Set-up and realisation

The 17α-Ethinylestradiol degradation experiments were carried out from June to August 2000. In week 6 the DV outlet of WWTP 1 was blocked for a time. In weeks 6 and 8 uncontrolled denitrification and floating sludge appeared in the SP of WWTP 2. Therefore, no sludge sampling was done in week 6.

The debit influent EE2 concentration amounted to 0.1 µg/l.

The hydraulic detention time of the synthetic wastewater in WWTPs 1 and 2 amounted to 13.9 – 14.5 and 13.1 – 13.6 h, resp.

Corollary parameters

The effluent nitrate-N concentrations exceeded the target value by about 50 %. All other target values were met except those of nitrate-N and ammonia-N in week 9, WWTP 1 (see tables 1, 40 to 50). Nevertheless, the experiments formally must be estimated as failed.

Concentrations

In week 6 no EE2 determinations were made at all because of the problems in the experimental realisation (see **above**).

Beside of this, EE2 determinations obviously failed in some cases. For example, the influent EE2 concentration of WWTP 1 was determined n.d. in week 5, 8, and 10 (see table 51).

The EE2 concentrations (see tables 51, 52) were calculated as MEAN of two determinations, respectively. The two values frequently considerably differed one from the other. For this reason also, the calculated data are only hardly useful. An interpretation is only possible under fundamental restrictions.

Again, the varying influent EE2 concentrations probably were caused by the performance of the peristaltic pumps.

When concentrations could be determined and calculated, in many cases the data are inconsistent. E.g., an increase of both the liquid and the solid phase EE2 concentration in the AV was determined for WWTP 2 in week 8. The dry matter concentrations in the DV and the AV were very similar (see tables 46, 47). Since the DV effluent is the only influent to the AV, an increase of both the water and the sludge EE2 concentration in the AV **can be excluded**.

Mass balances

EE2 mass balances could not be calculated because of the little number of consistent EE2 data and due to the effluent nitrogen-N concentrations distinctly exceeding the target value.

1.4. Bisphenol A degradation experiments

Set-up and realisation

The BPA degradation experiments were carried out from May to July 2001. The debit influent BPA concentration amounted to 10 µg/l.

The hydraulic detention time of the synthetic wastewater in WWTPs 1 and 2 amounted to 15.9 – 16.9 and 16.4 – 16.7 h, resp.

Corollary parameters

All corollary parameters met the guideline target values (see tables 1, 4 to 14). The sludge dry matter concentration increased from 1.79 to 2.63 g/l in week 3 to 2.83 to 3.45 g/l in week 8. Thus, the experiments in general were successful.

Concentrations

The BPA **determinations** started at the beginning of week 3 when the corollary analytical parameters showed that both WWTPs run stabile.

Both WWTPs showed an exhaustive reduction of the water phase BPA. The overall elimination rate of WWTP 1 and 2 increased by time from ~ 56 and ~ 73 % to ~ 98 %, respectively (see tables 15, 16). A strong reduction already appeared in the denitrification vessels where the water phase BPA concentration $c_1(\text{BPA})$ decreased from ~ 4 µg/l in week 3 to 0.38 and 0.24 µg/l in week 8, respectively (figures 5, 6). Reduction of water phase BPA was further extended in the aeration vessels where $c_1(\text{BPA})$ decreased by time from 3.5 and 2.6 µg/l, respectively, below the limit of detection (LOD). So, after 8 weeks these two-stage treatment systems were capable to **exhaustively** eliminate BPA from the liquid phase. Though $c_1(\text{BPA})$ was not detectable in the AV in week 8 the effluent concentrations amounted to 0.22 and 0.28 µg BPA/l, respectively. Desorption from excess sludge must have taken place during its short detention in the separator. If not stated otherwise, the values of week 8 are consecutively discussed.

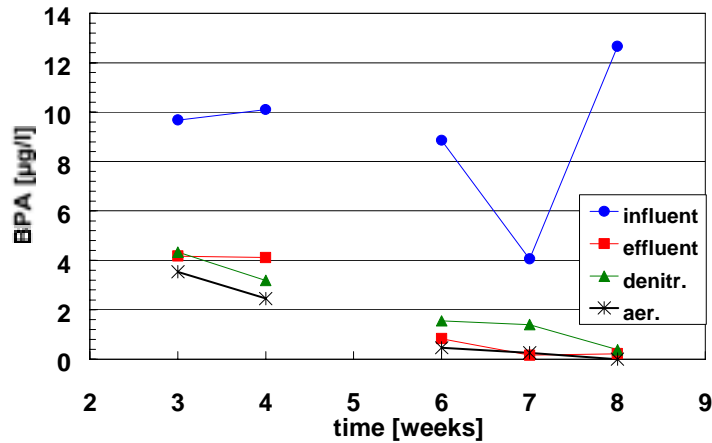


Figure 5: Water phase BPA concentrations of WWTP 1

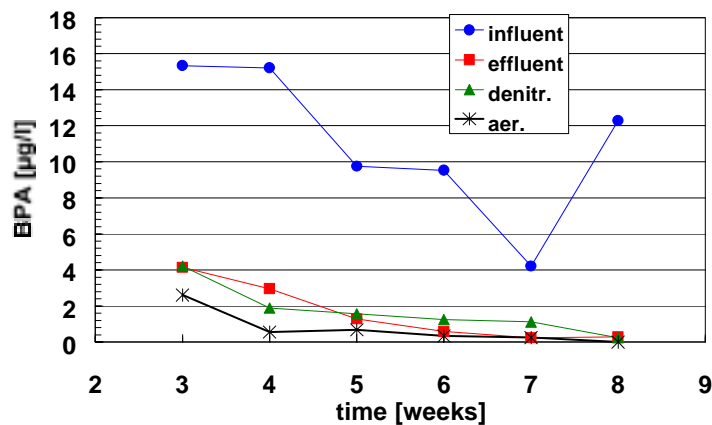


Figure 6: Water phase BPA concentrations of WWTP 2

Obviously, the lowest $c_i(\text{BPA})$ possible was only scarcely dependent on the influent BPA concentration that occasionally differed from the debit concentration of $10 \mu\text{g/l}$ due to varying performances of the peristaltic pumps. In week 7 the influent solution obviously was made at only half of the debit. In fact it depended on time and thus on the adaptation ability of the bacteria. The varying influent $c_i(\text{BPA})$ also hardly influenced the sludge BPA concentration $c_s(\text{BPA})$. Over time $c_s(\text{BPA})$ of WWTP 1 and 2 run very similar **independent on the sometimes very different influent concentrations**. The slight increase of $c_s(\text{BPA})$ in week 8 could have been caused by the increased influent $c_i(\text{BPA})$.

$C_s(\text{BPA})$ of both WWTPs also decreased by time: in the denitrification vessels from $\sim 1,100$ and $\sim 1,200 \mu\text{g/kg DM}$ in week 3 to $\sim 400 \mu\text{g/kg DM}$ in week 8, and in the aeration vessels from $1,100$ and 770 to $\sim 200 \mu\text{g/kg DM}$, respectively. For unknown reasons, no BPA was eliminated from sludge in the aeration vessel of WWTP 1 until week 5.

After aerobic elimination has started in WWTP 1 during week 5, the BPA concentrations immediately reached values very similar to those of WWTP 2.

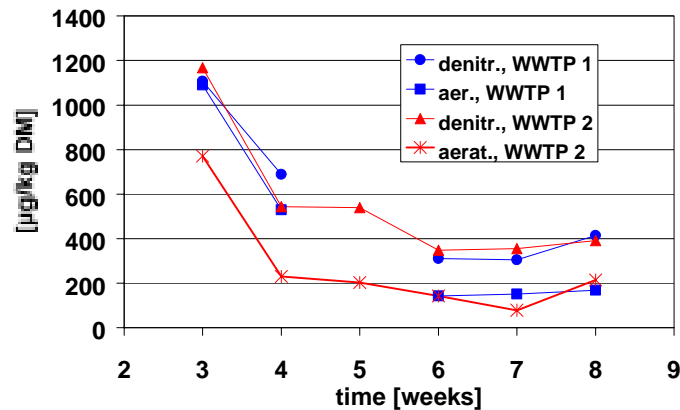


Figure 7: Solid phase BPA concentrations of WWTP 1 and 2

Mass balances

The denitrification stage influents consisted of the system influents carrying only dissolved BPA and the two recirculation fluxes, respectively (see figure 1). Both $c_s(\text{BPA})$ of the two circulating mass fluxes were lower than $c_s(\text{BPA})$ in the denitrification vessels. Thus, adsorption of influent BPA to sludge can be stated. The BPA elimination in the aerated stages must have been caused by degradation (or transformation). Both $c_s(\text{BPA})$ and $c_l(\text{BPA})$ in the aeration vessels were lower than in the denitrification vessels. $C_s(\text{BPA})$ could not be reduced below the LOD, neither after denitrification nor after aeration. Possible causes are: i) The sludge age of 21 days was too short for exhaustive degradation even though BPA was found to be readily degradable under aerobic conditions. ii) A certain portion of the BPA was permanently detracted from degradation when adsorbed to sludge in the denitrification vessels.

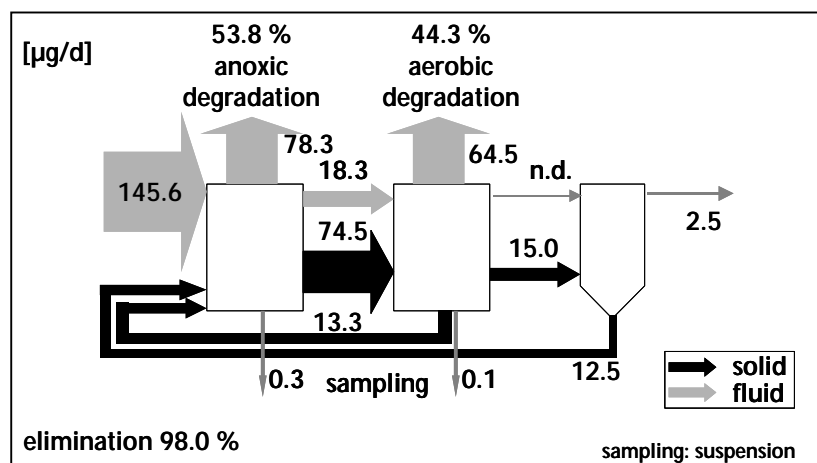


Figure 8: Bisphenol A mass balance of WWTP 1, week 8; elimination rates in relation to system AD+PW influent; all values [µg/d] except otherwise stated

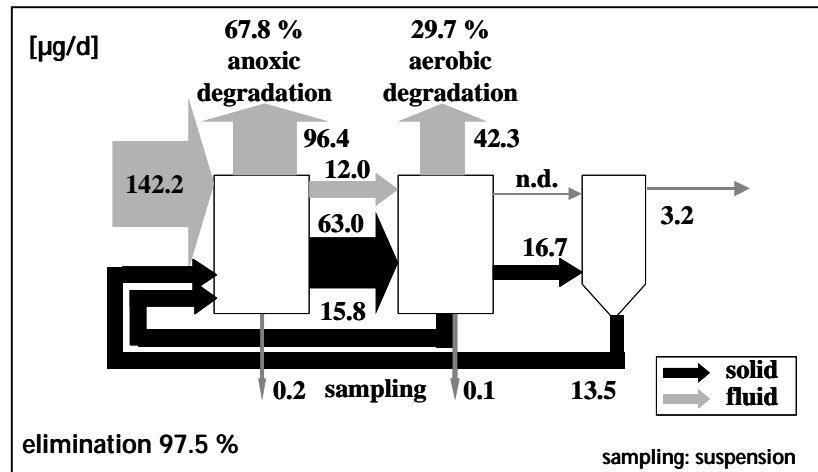


Figure 9: Bisphenol A mass balance of WWTP 2, week 8; elimination rates in relation to system AD+PW influent; all values [µg/d] except otherwise stated

The mass balances of both WWTPs show good similarity even though some differences occur (see tables 19, 20, figures 8, 9). The BPA elimination resulted from anoxic degradation in the denitrification vessels, and aerobic degradation in the aeration vessels. Initial adsorption during denitrification amounted to 49 and 34 µg/d (33 and 24 % of system influent BPA mass flux, respectively). The solid-phase bound BPA mass flux calculated also could be the sum of the converse effects of adsorption to sludge and degradation of adsorbed BPA. Thus, the figures represent the lower limit of the anoxic adsorption rate. Anoxic degradation can be calculated to 54 and 68 % of system influent BPA, respectively. After denitrification, $c_s(\text{BPA})$ was further reduced in the aeration vessels by ~ 200 µg/kg DM (~ 50 %) resulting in a decrease of the solid phase BPA mass flux by 70 and 56 %, respectively. Dissolved BPA was completely degraded in the aeration vessels. Thus, aerobic degradation amounted to 44 and 30 % of system influent BPA, respectively.

Since $c_l(\text{BPA})$ was reduced in the aeration vessels below the LOD no dissolved BPA subsequently flowed into the separators. The separators were not aerated and the effluent oxygen concentrations amounted to 0.3 to 0.6 mg O₂/l. The sludge deposits at the bottom of the separators probably became even anaerobic. This is the case in full-scale WWTPs as well. It can be assumed that a new equilibrium was formed during separation by BPA desorption from sludge. This desorption caused effluent mass fluxes of 2.5 and 3.2 µg/d, respectively. The anoxic degradation calculated for clarification is a cumulative error value of these mass balances representing dry matter loss with effluent, data variability, the inexact estimation of the dry matter concentration of the excess sludge, etc. This is not the case for the BPA degradation experiments because the IR mass fluxes was calculated as the difference between SP influent and total system effluent, resp. Thus, the estimated IR sludge BPA concentration was lower than the AV sludge BPA concentration.

1.5. Nonylphenolmonoethoxylate and nonylphenoldiethoxylate degradation experiments

Set-up and realisation

The nonylphenolmonoethoxylate (NP₁EO) and nonylphenoldiethoxylate (NP₂EO) degradation experiments were carried out from November 2001 to January 2002. Because of the very high costs of commercially available nonylphenoethoxylate (NPEO) standard solutions, Imbentin 20 was used for supplying NP₁EO and NP₂EO to the WWTPs. Imbentin 20 (Imb. 20) is a technical mixture of NP₁EO and NP₂EO. Since the exact concentrations of NP₁EO and NP₂EO are not given by the producer, they must be quantified by means of single standards.

The debit Imb. 20 influent concentration amounted to 1.9055 mg/l.

No sampling was carried out in week 7, WWTP 2, because the IR pump didn't work for a time and all parts of the WWTP became anaerobic. For unknown reasons, little amounts of floating sludge frequently appeared in separators of both WWTPs. The floating sludge was held back before the separator outlets.

Corollary parameters

The corollary parameters DOC and COD elimination rate, temperature, and pH-value met the target values (see tables 57 to 67). The dry matter concentrations and the inorganic nitrogen parameters met the target values until week 8 or 9 except the nitrate-N concentration in week 6, WWTP 2. In the latter case, the data are inconsistent. Possibly the samples or values were mixed up.

Concentrations

The introduction of a normal phase HPLC method for the determination of NPEO failed until February 2000. Therefore, a GC/MS method for the determination of NPEO should be developed. Since the introduction of this GC/MS method is not yet completed, the first samples were analyzed for NP₁EO and NP₂EO but the concentrations values could not yet be calculated.

2. Fate of EDC in full-scale WWTP – field samplings

Field samplings were done in co-operation with a German research project (02WA9979) on EDCs in wastewater treatment and carried out by the Technical University of Berlin, Department of Water Technology. The schematic diagram of the technology applied at WWTP 6 is shown in figure 10.

Two main differences appear to the set-up and realisation of the laboratory experiments. At first, denitrification and aeration are in addition followed by phosphorus elimination. The intern reflux of excess sludge is lead into the P-elimination tanks. Second, the liquor of the sludge dewatering is returned to the influent before the screen.

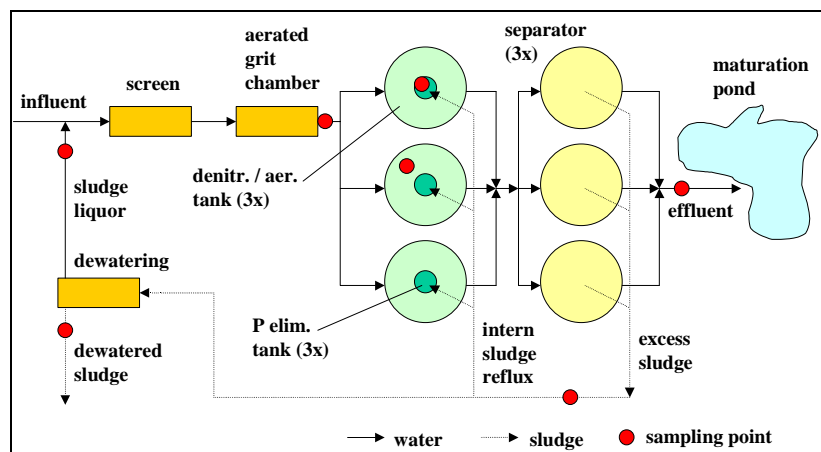


Figure 10: Schematic diagram of the technology applied at WWTP 6

2.1. Sampling 2000-05-26

An early sampling was carried out within the frame of the German research project on 2000-05-26. Samples of influent, effluent, excess sludge, and dewatered sludge were drawn and analyzed for NP, OP, BPA, EE2, and E2.

All E2 values were n.d. or not calculable (n.c.) (see table 68). EE2 was detectable only in sludge. Its concentrations amounted to 1 µg/kg DM in the excess sludge and to 10 µg/kg DM in the dewatered sludge. The concentrations of the three industrial xenoestrogens amounted to 0.36 to 1.27 µg/l in the water phases and to 150 to 1240 µg/kg DM in sludge. All NP, OP, and BPA data were lower than the MEAN of 4 to 17 samplings in municipal German WWTP (WELTIN ET AL., 2002; GEHRING ET AL., 2002). A distinct overall elimination only appeared for BPA (71.7 %), while an increase of the NP concentration was observed (35.9 %).

2.2. Sampling 011127sm

Seven different samples were drawn on 2001-11-27 (see figure 10). Calculating mass balances is made more complicated because two important samples could not be taken due to the technical conditions: the sample of the WWTP influent before the inlet of the sludge liquor and the sample of the P-elimination effluent (separator influent). Samples were (are) analyzed for all the 8 EDC listed in chapter 1.1.

Several problems appeared during preparation and analysis of the liquid samples. The blanks in part were very high, thus unrealistic, and not reproducible. The concentrations of the steroids were not calculable because of problems with the time window of the certain internal standard (peaks cut off). The NP_{1,2}EO concentrations were not calculable because the corresponding GC/MS method is not yet completely introduced. The blanks of the steroids were unusual high as well. For all these reasons, sample preparation and EDC determinations were stopped until the problems were solved. The solids have been prepared meanwhile but not yet analyzed for EDC.

The liquids were analyzed for EDC but since high uncertainties exist when interpreting the data the determinations have to be repeated. The concentrations given in table 69 are corrected by the respective blank but LOD and LOQ were not taken into consideration.

All influent concentrations of the xenoestrogens NP, OP, and BPA were n.d. even in the extracts. That is very unlikely and possibly a hint on an analytical problem.

The OP concentrations are about one order of magnitude lower than the NP concentration. This relation is found in many cases and well-known from other sampling campaigns and from the literature.

The water phase BPA concentration in the aeration tank is lower than in the denitrification tank. This finding agrees with the results of the laboratory experiments (see chapter 1.4.).

The NP, OP, and BPA effluent concentrations were distinctly higher than the respective water phase concentration of the last activated sludge tank (phosphorus elimination). This is a quite unusual phenomenon and has to be confirmed.

3. Comparison of the behaviour of EDC in real WWTP and lab scale plants

3.1. 17β -estradiol degradation

The data derived from the laboratory experiments are incomplete and in part inconsistent. The progress of the E2 elimination with time cannot be estimated. The calculated mass balance for WWTP 2, week 8, and the little E3 data show that E2 is degraded to E1 to a large extent already in the DV (anoxic conditions). E1 is degraded to E3 in the AV (aerobic conditions). The degradation of E1 cannot be estimated.

At this moment, no final conclusions can be made. Two additional laboratory experiments were made from July to September 2001. A mixture of E2, EE2, BPA, M, NP, and OP was used. Because of technical problems regarding the GC/MS the samples have been prepared for determination but not yet analyzed for EDCs. Additional field samplings were not possible due to the high costs i.e. of the sample preparation procedure. The repetition of the GC/MS analysis of the liquid samples from WWTP 6, the first analysis of the solids from WWTP 6, and the analysis of the degradation experiments of the analyte mixture are just running.

3.2. 17α -ethinylestradiol degradation

No conclusions are possible at all. The data are consistent, a number of samplings and determinations failed.

3.3. Bisphenol A degradation

In the laboratory, water phase BPA is distinctly degraded already in the DVs (anoxic conditions). The degradation was completed in the AVs even below the LOD. In the DVs, adsorption of BPA to sludge also appeared. A certain portion of this BPA adsorbed was detracted from degradation within the sludge age and hydraulic detention time applied. This BPA adsorbed led to desorption in the SPs.

The quality of the field data is somewhat better than in the case of the steroids but not sufficient. Water phase BPA was distinctly lower in the AV than in the DV.

3.4. Nonylphenolmono- and –diethoxylate degradation

Since the development of a method for the determination of NPEOs is not yet successfully completed, the chromatograms recorded by GC/MS could not yet be quantified. No conclusions can be made about the NPEO degradation neither in the laboratory experiments nor in the full-scale WWTP no. 6.

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IV. DEVIATIONS FROM TECHNICAL ANNEX AND REASONS

The subcontractors GEW and SCREEN TEC included in the project program renounced to participate in the project.

V. CONCLUSIONS

The common but scarcely applied wastewater treatment technology of simultaneous aerobic sludge treatment is well suited to reduce the water phase BPA concentration below the MDL of 0.1 ng BPA/l (25 pg BPA). Release of BPA from sludge during clarifying can lead to newly increased effluent concentrations. The aeration of the separator, i.e. of the sludge deposit, a sludge detention time in the separator as short as possible, and an adequate hydraulic detention time of the water in the aeration stage possibly can improve the BPA elimination efficiency of aerobic wastewater treatment.

Simultaneous aerobic sludge treatment also should be sufficient for the exhaustive elimination of E2. But in this case additional experiments have to be carried out. I.e. the formation and degradation of E3 still has to be studied.

Sampling, transportation, and sample preparation were much more expensive than calculated before. This has to be taken into consideration for future research.

If samples must be sent abroad they should be held frozen all the time. Since this didn't work at all within this project, transportation of water samples should be restricted as far as possible. Water samples should be extracted by SPE, dried in a nitrogen stream, and then shipped as loaded SPE cartridges in a nitrogen atmosphere. Solid samples should be sent freeze-dried. Since many commercial plastics contain either bisphenol A or tetrabromobisphenol A or even are made from bisphenol A monomers (polycarbonate, epoxy resins), plastics totally has to be excluded from sampling, sample storage, sample preparation, and analytical procedures.

One or two field samplings are not sufficient in order to estimate the fate of EDCs in a full-scale WWTP. As discussed in the literature, distinct differences appear from month to month, from week to week, and even from day to day (influent EDC concentration!). Highest attention has to be paid to sampling locations, sample volume, sample homogenisation, etc. I.e. samples of all important volume fluxes has to be drawn.

VI. Glossary

AD	analyte and diet solution	SPE	solid phase extraction
AE	aeration, aeration unit	T	temperature
aer.	aeration, aeration vessel	TO	temperature and oxygen
AV	aeration vessel	WWTP	wastewater treatment plant
BPA	bisphenol A		
$c_l(X)$	liquid phase concentration of compound X		
COD	chemical oxygen demand		
cond.	Conductivity		
$c_s(X)$	solid phase concentration of compound X		
denitr.	denitrification, denitrification vessel		
DIN	Deutsches Institut für Normung		
DM	dry matter		
DOC	dissolved organic carbon		
DV	denitrification vessel		
E1	Estrone		
E2	17 β -estradiol		
E3	Estriol		
EDC	endocrine disrupting compound / chemical		
EE2	17 β -ethinylestradiol		
EF	Effluent		
GC/MS	gas chromatography / mass spectrometry		
GPC	gel permeation chromatography		
HPLC	high performance liquid chromatography		
Imb. 20	Imbentin 20		
IR	intern reflux of excess sludge		
LOD	limit of detection		
LOQ	limit of quantification		
M	Mestranol		
MDL	method detection limit		
N	Nitrogen		
n.c.	not calculable		
n.d.	not detectable		
n.m.	not determined		
n.q.	not quantifiable		
NP	4-nonylphenol		
NP _{1,2} EO	nonylphenolmono- and -diethoxylate		
NP ₁ EO	nonylphenolmonoethoxylate		
NP ₂ EO	nonylphenoldiethoxylate		
NPEO	nonylphenolethoxylates		
OECD	Organization for Economic Cooperation and Development		
OP	4-tert-octylphenol		
P	Phosphorus		
PW	potable water		
RA	denitrification reflux of activated sludge		
SA	sample, sampling		
SEC	size exclusion chromatography		
SP	Separator		

ANNEX I

Publications:

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ANNEX II (informative)

Analytical methods:

Silanisation of Glass Ware

Due to their moderate hydrophilicity the endocrine disruptors (EDC) can adsorb to glass surfaces. Therefore, inactivation of glass ware by silanisation is advised. Long contact times during sample preparation and the usually very low concentrations of EDC in the samples require the preventive treatment of all glass equipment used.

For silanisation the glass ware is stored in 1 N HCl for at least 12 hours, rinsed with deionised water, air-dried and moistened by a solution of 5 % dimethyldichlorosilan (DMDCS, Supelco, Bellefonte, PA, USA) in toluene (v/v). After 10 to 15 s the glass surface is cleaned twice by toluene (p.a.) and three times by methanol (HPLC grade). Finally, the glass ware is dried in a stream of N₂.

Before silanisation becomes necessary again the glass ware is cleaned only by means of acetone (p.a.) and deionised water. Dishwashers as well as brushes must not be used due to the sensitivity of silanisation to mechanic stress. Silanisation has to be repeated after 4 to 5 cycles.

Activation and Conditioning of Silica Gel

For activation, silica gel (70 – 230 mesh, Merck, Darmstadt, Germany) is annealed for 6 hours at 600°C, mixed with 1.5% deionised water (v/v), and dried under occasional shaking for 12 hours at 60°C. The activated silica gel is stored in an exsikkator.

1 g is slurried in n-hexane:acetone 60:40 (v/v), filled into a glass column (V = 8 ml, Merck, Darmstadt, Germany), and conditioned by 10 ml of solvent, respectively.

Conditioning of SPE Columns

SPE columns (V = 6 ml) contain a upper non-polar phase of 200 mg LiChrolut RP 18 and a lower polar phase of 100 mg LiChrolut EN (Merck, Darmstadt, Germany). Conditioning is made by eluting the column bed in succession with 3 x 2 ml n-hexane, 2 x 2 ml acetone, 3 x 2 ml methanol, and 5 x 2 ml deionised water (pH = 3). Glass wool annealed and surface-sealed by silanisation is placed above the RP 18 phase in order to filter fine particles that still might remain in the liquid sample even after centrifugation.

Conditioning of GPC Columns

24 hours before use for gel permeation chromatography (size exclusion chromatography) Bio-Beds[®] S-X3 Beads gel (Bio-Rad Laboratories, Hercules, CA, USA) is slurried in dichloromethane:cyclohexane 50:50 (v/v) and stored for swelling. After 24 hours approximately 10 g of the gel are filled into a glass column 300 mm in length and 28 mm in diameter. The gel bed is covered by a layer of quartz sand (corn size 1 mm, h = 10 mm) and conditioned with 50 ml of solvent. GPC columns can be used up to 50 times.

Preparation of Liquid Samples

An aqueous sample (V = 0.2 to 1 l) is separated by centrifugation for 20 min at 29,000 x g and 4°C (Beckman J2-MC, Palo Alto, CA, USA). If necessary, centrifugation is repeated up to three times.

After acidification to pH = 3 (2 M HCl) and addition of 25 ng of the internal standards 4n-nonylphenol (Riedel-de Haën, Seelze, Germany), bisphenol-A-d₁₆ (Aldrich Chemicals,

Milwaukee, WI, USA), and 17 β -ethinylestradiol-17-acetate (Sigma-Aldrich Chemicals, Steinheim, Germany), respectively, the sample is stirred for 60 min.

By means of a vacuum extraction unit, the sample then is sucked through the solid phase extraction column prepared as described above at a flow of 5 – 10 ml/min. After drying of the column (N₂-stream, 60 min) the enriched EDC are eluted by 6 x 1 ml acetone. The acetone extract again is dried in a nitrogen stream.

Preparation of Solid Samples

A portion of 1 to 2 g of freeze-dried and homogenised sewage sludge or other solid material is filled into a glass fibre thimble. Internal standards (1 μ g of 4n-nonylphenol, bisphenol-A-d₁₆, 17 β -estradiol-17-acetate, respectively) are added to 90 ml of methanol are added. The spiked sample is extracted by Soxhlet-extraction (6 h), reduced to approximately 5 ml by rotary evaporation (Laborotor 4002, Heidolph, Kehlheim, Germany) and dried (N₂ stream).

The dry residue is collected in dichloromethane:cyclohexane 50:50 (v/v) and transferred to the gel permeation column prepared as described above. The Soxhlet glass flask is additionally rinsed at least twice using 1 ml solvent, respectively. After the sample sunk into the column bed, solvent is added carefully flow. Attention has to be paid that the column must not fall dry at any moment. The undesired matrix passes through the column within the first 80 to 85 ml of the collected sample. The EDC follow in the second fraction (80/85 to 180 ml). The fraction volumes can slightly differ along with variations in solvent addition. The fraction of interest is reduced to 0.5 ml by rotary evaporation and dried in a N₂-stream. The column is cleaned by running additionell 50 ml of solvent.

Silica Gel Clean-up

The dried SPE and GPC residues are taken up by 3 x 200 μ l of acetone and then supplied to a silica gel column activated as described above. After sinking into the column bed, most of the remaining matrix adsorbs to the silica gel and the cleaned extract including the EDC is eluted from the column by 7.1 ml of hexane:acetone 60:40 (v/v). Again, the extract is dried in a N₂-stream.

Derivatization

50 μ l of the silylisation chemical (Sylon BTZ, Supelco, Bellafonte, PA, USA) are added to the dry residue. The derivatisation takes place at 60°C for 30 min. After cooling down to room temperature, surplus Sylon BTZ is deactivated by adding 50 μ l of deionised water (pH = 3). The derivative is extracted by shaking with 250 μ l (liquid original samples) or 1000 μ l (solid original samples) toluene/Mirex solution (1 μ g Mirex/l), respectively. Mirex (Institute of Organic Industrial Chemistry, Poland) serves as a volume correction standard. Phase separation is optimised by centrifugation (3 min, 5,000 g, 4°C). The toluene phase then is transferred into a micro-vial by means of a Pasteur pipette.

GC/MS Determination

A gas chromatographic system GC HP 6890 equipped with a phenyl-methyl-siloxane column (HP-5MS, 5%, 30 m x 200 μ m x 0,25 μ m nominal) coupled with a high resolution mass-spectrometer MSD HP 5973 (Hewlett Packard, Boeblingen, Germany, respectively) is applied for the analysis.

Helium is used as carrier gas at a flow of 1.2 ml/min.

By autosampling, 1 μ l for alkylphenol determination and 10 μ l for steroid and bisphenol-A determination is supplied to a low temperature injector Cryofoc 504 (Gerstel, Mülheim/Ruhr, Germany).

Injector program: 45°C/2 min/isotherm, split 1:20, 12°C/min to 280°C, splitless 0.5 min, 12°C/s to 300°C, post-run 300°C/2 min/isotherm

Oven program: 120°C/2 min/isotherm, 10°C/min to 280°C, 280°C/2 min/isotherm, post-run 300°C/10min/isotherm

The single-ion-modus (SIM) target and qualifier ions are given in table 3, the photo multiplier voltage is set to 2,800 V.

Examination is made by means of Enhanced ChemStation G1701BA software, V. B.00.00.

Table 3: GC/MS Sampled Target and Qualifier Ions of the Analytes

Analyte		OP	NP	4n-NP	BPA	BPA-d ₁₆	E1	E2	E2-acet	E3	EE2	M
Target	m/z	207	207	179	357	368	342	416	386	345	440	367
Qualifier	m/z	208	208	178	358	367	257	417	387	504	425	227
		191	221	292	372	386	218	285	244	311	285	242
		-	-	-	-	-	-	286	-	-	-	-
OP	4-tert-octylphenol		BPA-d ₁₆	bisphenol A-d ₁₆	EE2	17 α -ethinylestradiol						
NP	4-nonylphenol			E1	estrone		M	mestranol				
4n-NP	4n-nonylphenol			E2	17 β -estradiol							
BPA	bisphenol A			E2-acet	17 β -estradiol-17-acetate							

ANNEX III (informative):

Originalata determined and values calculated

Table 4: Influent WWTP 1; corollary parameters BPA degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μs/cm]	
2001-03-06	<0.1	0.495	7.77	182.70	400	7.80	403	
2001-03-12	<0.1	0.360	15.38	138.70	310	7.47	470	
2001-03-19	<0.1	<0.1	15.27	142.40	310	7.55	408	
2001-03-27	<0.1	0.290	13.78	109.10	220	7.45	392	
2001-04-02	<0.1	0.340	26.70	175.40	440	7.41	430	
2001-04-09	<0.1	<0.1	24.75	150.50	410	7.55	480	
2001-04-17	<0.1	<0.1	21.11	148.60	340	7.58	506	
2001-04-23	<0.1	0.045	24.01	157.90	370	7.65	582	
2001-05-02	0.34	0.920	15.11	175.50	400	7.70	483	
2001-05-07	<0.1	2.400	23.89	163.10	370	7.67	628	
2001-05-14	<0.1	1.160	22.60	168.90	390	7.51	598	
2001-05-21	<0.1	1.48	32.11	219.10	540	7.41	695	3
2001-05-28	<0.1	2.98	20.55	169.20	390	7.53	585	4
2001-06-08	<0.1	1.360	15.90	195.10	370	7.53	517	5
2001-06-11	<0.1	0.967	18.50	136.60	370	7.77	542	6
2001-06-18	<0.1	1.350	22.20	136.85	360	7.78	491	7
2001-06-25	<0.1	2.160	16.75	141.80	360	7.28	408	8

Table 5: Effluent WWTP 1; corollary parameters BPA degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[µs/cm]	
2001-03-06	2.380	0.590	2.458	6.510	44	7.66	654	
2001-03-12	5.750	0.790	19.160	6.293	36	7.57	424	
2001-03-19	6.240	1.690	0.428	5.880	39	7.42	417	
2001-03-27	0.620	8.820	0.152	5.410	32	6.94	403	
2001-04-02	0.500	12.290	0.222	7.350	40	7.12	410	
2001-04-09	0.391	13.270	0.240	8.050	37	7.21	423	
2001-04-17	0.780	10.520	0.980	9.880	38	7.34	412	
2001-04-23	0.583	13.010	0.465	10.570	48	7.22	423	
2001-05-02	1.887	9.430	6.650	19.940	83	7.43	479	
2001-05-07	2.490	7.940	3.200	13.290	61	7.46	480	
2001-05-14	0.415	10.760	1.390	11.310	54	7.35	444	
2001-05-21	0.706	11.6	0.67	9.5	45	7.34	430	3
2001-05-28	1.65	8.83	2.55	10.5	53	7.46	484	4
2001-06-08	1.820	9.750	2.810	7.000	43	6.77	439	5
2001-06-11	0.610	10.400	0.260	6.850	35	7.03	410	6
2001-06-18	0.225	12.200	0.245	5.800	24	6.98	410	7
2001-06-25	0.130	13.400	0.195	5.550	28	6.79	391	8

Table 6: Denitrification vessel WWTP 1; corollary parameters BPA degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[µs/cm]	
2001-03-06	0.319	0.500	39.98	45.95	132	2988	7.50	653	
2001-03-12	1.090	<0.1	26.56	39.60	117	3310	7.54	586	
2001-03-19	0.890	0.230	10.92	33.55	106	3282	7.50	465	
2001-03-27	<0.1	0.160	8.25	30.76	96	3422	7.29	437	
2001-04-02	0.200	1.900	7.81	11.63	59	3190	7.23	463	
2001-04-09	<0.1	0.620	9.42	15.26	49	3214	7.25	475	
2001-04-17	0.370	1.460	8.35	15.45	51	1776	7.23	457	
2001-04-23	0.229	1.950	7.80	18.53	63	2210	7.29	471	
2001-05-02	0.553	1.340	15.99	27.46	100	1906	7.29	518	
2001-05-07	<0.1	0.820	9.86	25.45	85	1562	7.42	515	
2001-05-14	<0.1	2.050	8.82	20.84	75	3190	7.30	487	
2001-05-21	<0.1	1.79	8.71	18.2	62	2606	7.21	476	3
2001-05-28	<0.1	1.76	11.47	18.46	65	2646	7.31	538	4
2001-06-08	<0.1	1.370	10.40	12.40	58	2738	7.39	488	5
2001-06-11	<0.1	1.035	10.35	13.55	48	2482	7.38	471	6
2001-06-18	<0.1	1.210	10.35	13.20	45	3056	7.32	478	7
2001-06-25	<0.1	2.100	10.00	11.80	42	3768	6.99	458	8

Table 7: Aeration vessel WWTP 1; corollary parameters BPA degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[µs/cm]	
2001-03-06	1.30	1.51	38.82	30.5	103	2992	7.52	655	
2001-03-12	4.41	0.47	22.08	28.2	90	3614	7.35	546	
2001-03-19	6.56	1.88	4.48	25.5	83	2980	7.07	421	
2001-03-27	0.54	6.37	1.42	26.8	83	2992	6.88	387	
2001-04-02	0.32	11.90	1.75	9.2	51	3244	6.91	406	
2001-04-09	<0.1	11.74	1.30	9.9	45	2580	6.85	427	
2001-04-17	0.74	9.56	1.64	10.3	44	1576	7.00	415	
2001-04-23	0.56	11.57	1.53	12.1	50	1800	6.91	425	
2001-05-02	1.65	9.76	8.78	22.2	91	1450	6.95	486	
2001-05-07	2.20	7.26	3.67	16.8	62	1078	7.11	475	
2001-05-14	0.55	10.52	1.99	14.2	56	2480	6.89	455	
2001-05-21	0.94	10.50	1.73	13.1	53	2206	6.95	441	3
2001-05-28	1.59	8.11	3.52	12.8	57	1810	7.09	489	4
2001-06-08	1.65	8.95	2.95	8.0	51	2262	6.87	435	5
2001-06-11	0.55	8.81	1.55	7.1	32	2536	6.82	418	6
2001-06-18	0.28	10.02	1.77	7.1	30	3382	6.74	415	7
2001-06-25	0.47	10.50	1.10	7.0	31	3522	6.44	396	8

Table 8: Influent WWTP 2; corollary parameters BPA degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[µs/cm]	
2001-03-06	<0.1	0.50	7.77	183	400	7.80	403	
2001-03-12	<0.1	0.36	15.4	139	310	7.47	470	
2001-03-19	<0.1	<0.1	15.3	142	310	7.55	408	
2001-03-27	<0.1	0.29	13.8	109	220	7.45	392	
2001-04-02	<0.1	0.34	26.7	175	440	7.41	430	
2001-04-09	<0.1	<0.1	24.8	151	410	7.55	480	
2001-04-17	<0.1	<0.1	21.1	149	340	7.58	506	
2001-04-23	<0.1	0.05	24.0	158	370	7.65	582	
2001-05-02	0.34	0.92	15.1	176	400	7.70	483	
2001-05-07	<0.1	2.40	23.9	163	370	7.67	628	
2001-05-14	<0.1	1.16	22.6	169	390	7.51	598	
2001-05-21	<0.1	1.48	26.3	172	390	7.66	598	3
2001-05-28	<0.1	2.88	19.2	155	350	7.50	569	4
2001-06-08	<0.1	1.16	22.5	150	330	7.77	518	5
2001-06-11	<0.1	1.55	18.3	127	340	7.78	470	6
2001-06-18	<0.1	1.67	23.2	136	370	7.75	516	7
2001-06-25	<0.1	1.73	18.3	138	350	7.24	421	8

Table 9: Effluent WWTP 2; corollary parameters BPA degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[µs/cm]	
2001-03-06	1.00	0.97	44.4	7.2	45	7.86	728	
2001-03-12	1.57	0.54	45.2	7.2	34	7.86	481	
2001-03-19	6.73	0.64	26.4	4.2	30	7.85	576	
2001-03-27	3.89	6.44	0.36	5.4	37	6.84	417	
2001-04-02	0.24	11.3	0.22	8.5	45	7.28	408	
2001-04-09	0.39	11.4	0.19	5.5	33	7.21	408	
2001-04-17	0.39	11.4	0.23	5.7	34	7.20	374	
2001-04-23	0.53	13.3	0.18	6.4	36	7.02	406	
2001-05-02	0.59	7.65	13.9	14.1	64	7.63	539	
2001-05-07	1.45	7.05	13.2	12.4	61	7.64	620	
2001-05-14	0.45	11.7	1.16	12.5	55	7.16	501	
2001-05-21	0.98	11.4	1.18	11.7	57	7.07	450	3
2001-05-28	1.82	9.67	1.83	10.4	53	7.28	478	4
2001-06-08	1.31	10.1	0.70	10.0	43	7.24	420	5
2001-06-11	1.18	10.8	0.63	8.0	35	6.92	419	6
2001-06-18	0.93	9.68	0.89	6.5	31	7.02	419	7
2001-06-25	0.82	12.2	0.37	6.3	28	6.79	393	8

Table 10: Denitrification vessel WWTP 2; corollary parameters BPA degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[µs/cm]	
2001-03-06	0.46	0.77	48.0	50.9	144	2958	7.50	723	
2001-03-12	0.74	<0.1	49.2	35.0	103	3452	7.52	659	
2001-03-19	1.04	0.20	26.3	28.5	83	3024	7.61	596	
2001-03-27	0.30	0.83	9.36	18.8	66	2888	7.27	474	
2001-04-02	0.16	1.51	8.89	16.1	73	2898	7.19	466	
2001-04-09	<0.1	1.79	8.84	11.9	46	3688	7.24	475	
2001-04-17	1.22	2.29	8.61	13.4	54	3282	7.22	451	
2001-04-23	<0.1	1.44	7.78	12.7	48	2714	7.23	467	
2001-05-02	0.90	1.19	26.5	27.3	105	2532	7.27	581	
2001-05-07	0.35	1.95	21.8	26.6	81	1448	7.53	632	
2001-05-14	<0.1	2.06	9.26	22.4	72	2332	7.24	488	
2001-05-21	<0.1	1.86	8.70	21.9	74	2294	7.26	481	3
2001-05-28	<0.1	1.85	9.62	20.9	76	2332	7.36	519	4
2001-06-08	<0.1	0.96	9.47	19.3	66	2728	7.40	467	5
2001-06-11	<0.1	1.63	9.85	12.9	48	2636	7.28	476	6
2001-06-18	<0.1	1.96	11.0	15.3	67	2502	7.26	479	7
2001-06-25	<0.1	1.55	11.8	12.7	43	3270	6.94	463	8

Table 11: Aeration vessel WWTP 2; corollary parameters BPA degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μs/cm]	
2001-03-06	1.15	10.9	0.74	13.2	56	1824	6.85	481	
2001-03-12	1.76	10.0	1.69	12.4	60	1764	6.99	482	
2001-03-19	1.46	9.29	1.33	12.1	52	2144	6.93	419	
2001-03-27	1.00	9.80	1.75	8.5	37	2350	6.77	423	
2001-04-02	0.21	8.55	0.25	10.7	55	3326	6.99	405	
2001-04-09	<0.1	9.89	1.24	7.2	37	3250	6.98	407	
2001-04-17	<0.1	10.3	0.31	8.7	37	3118	6.95	390	
2001-04-23	<0.1	12.7	0.57	8.6	35	2962	6.82	410	
2001-05-02	0.65	8.14	16.3	22.2	83	2134	7.21	547	
2001-05-07	1.09	5.89	18.6	15.7	58	1328	7.35	600	
2001-05-14	0.36	10.8	1.05	13.8	53	2036	6.90	441	
2001-05-21	1.15	10.9	0.74	13.2	56	1824	6.85	481	3
2001-05-28	1.76	10.0	1.69	12.4	60	1764	6.99	482	4
2001-06-08	1.46	9.29	1.33	12.1	52	2144	6.93	419	5
2001-06-11	1.00	9.80	1.75	8.5	37	2350	6.77	423	6
2001-06-18	0.89	8.87	2.30	8.3	37	2554	6.75	420	7
2001-06-25	0.51	9.95	2.18	7.6	31	3100	6.45	396	8

Table 12: pH-value and temperature WWTP 1 and 2; BPA degradation experiments

PH value		
n = 6	WWTP 1	WWTP 2
influent	7.55 (± 0.16; s = 0.20)	7.62 (± 0.17; s = 0.21)
denitrification	7.06 (± 0.23; s = 0.28)	7.25 (± 0.16; s = 0.16)
aeration (nitrification)	7.27 (± 0.12; s = 0.15)	6.79 (± 0.15; s = 0.19)
effluent	6.82 (± 0.18; s = 0.22)	7.05 (± 0.15; s = 0.19)
Temperature		
n = 28	WWTP 1	WWTP 2
aeration (nitrification)	21.5 (± 0.56; s = 1.5)	21.8 (± 0.56; s = 1.5)

Table 13: Hydraulic detention time WWTP 1 and 2; BPA degradation experiments

week	hydraulic detention time WWTP 1				hydraulic detention time WWTP 2			
	denitr.	aerat.	separator	total	denitr.	aerat.	separator	total
	[h]	[h]	[h]	[h]	[h]	[h]	[h]	[h]
3	1.53	1.53	1.96	16.2	1.53	1.53	2.14	16.5
4	1.46	1.46	1.90	15.9	1.47	1.47	1.90	16.4
5	1.50	1.50	2.01	16.9	1.48	1.48	1.92	16.7
6	1.50	1.50	1.77	16.4	1.45	1.45	1.87	16.6
7	1.51	1.51	1.88	16.8	1.45	1.45	1.87	16.5
8	1.50	1.50	1.88	16.7	1.46	1.46	1.88	16.6

Table 14: Sludge age WWTP 1 and 2; BPA degradation experiments

week	WWTP 1		WWTP 2	
	denitr.	aer.	denitr.	aer.
	[d]	[d]	[d]	[d]
3	20	20	20	20
4	21	21	21	21
5	21	21	21	21
6	21	21	21	21
7	21	21	21	21
8	21	21	21	21

Table 15: BPA concentrations WWTP 1

week	concentration							
	influent	denitr. liquid	denitr. solid	denitr. total	aer. liquid	aer. solid	aer. total	effluent
	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]
3	9.68	4.34	1,106	7.24	3.54	1,090	5.73	4.17
4	10.11	3.19	689.1	5.01	2.45	529.9	3.41	4.11
5	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
6	8.86	1.55	310.7	2.41	0.46	143.7	0.89	0.83
7	4.06	1.40	305.8	2.44	0.26	151.3	0.78	0.16
8	12.66	0.38	414.5	1.95	n.d.	168.7	0.59	0.22

Table 16: BPA concentrations WWTP 2

week	concentration							
	influent	denitr. liquid	denitr. solid	denitr. total	aer. liquid	aer. solid	aer. total	effluent
	[µg/l]	[µg/l]	[µg/kg DM]	[µg/l]	[µg/l]	[µg/kg DM]	[µg/l]	[µg/l]
3	15.33	4.21	1,168	6.91	2.61	770.1	3.99	4.15
4	15.21	1.89	544.5	3.16	0.55	231.2	0.96	2.96
5	9.76	1.57	539.5	3.01	0.68	202.4	1.13	1.29
6	9.53	1.25	348.7	2.15	0.34	143.4	0.69	0.58
7	4.21	1.12	354.7	2.14	0.25	78.68	0.47	0.23
8	12.28	0.24	391.8	1.52	n.d.	214.3	0.66	0.28

Table 17: Volume fluxes WWTP 1; BPA degradation experiment

week	volume flux									
	influent table water	influent diet / analyte solution	influent total	denitr. recircul.	intern recircul.	effluent denitr.	effluent aer.	sampling denitr. / aer. Resp.	effluent separator (calculated)	effluent (measured)
	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]
3	441	52.6	493.6	934	527	1.948	1.008	6.3	481	484
4	445	58.8	503.8	1.004	547	2.049	1.039	6.0	492	492
5	424	49.2	472.8	1.003	520	1.990	981	6.0	461	463
6	439	49.0	488.2	878	639	1.999	1.115	6.0	476	464
7	428	48.3	476.5	916	589	1.976	1.054	6.0	465	488
8	430	49	479	932	583	1.988	1.050	6.0	467	472

Table 18: Volume fluxes WWTP 2; BPA degradation experiment

week	volume flux									
	influent table water	influent diet / analyte solution	influent total	denitr. recircul.	intern recircul.	effluent denitr.	effluent aer.	sampling denitr. / aer. Resp.	effluent separator (calculated)	effluent (measured)
	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]
3	419	65.6	484.6	1.030	448	1.956	920	6.3	472	475
4	439	49.3	488.3	987	564	2.033	1.040	6.0	476	509
5	431	49.4	480.1	981	562	2.017	1.030	6.0	468	470
6	435	48.1	483.3	999	589	2.066	1.060	6.0	471	468
7	436	47.7	484.0	998	587	2.064	1.059	6.0	472	476
8	434	48	482	993	579	2.049	1.050	6.0	471	471

Table 19: BPA mass fluxes WWTP 1

week	mass flux										
	influent	effluent	denitr. recircul. liquid	denitr. recircul. solid	denitr. recircul. total	intern recirc. liquid	intern recirc. solid	intern recirc. total	denitr. samplin g liquid	denitr. samplin g solid	denitr. sampling total
	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]
3	114.71	48.42	79.28	49.06	128.3	44.73	52.95	97.68	0.65	0.44	1.09
4	122.19	48.58	59.11	23.11	82.2	32.21	23.92	56.12	0.46	0.26	0.72
5	n.m.	n.m.	n.m.	n.m.		n.m.	n.m.		n.m.	n.m.	
6	103.82	9.26	9.74	8.96	18.7	7.09	11.38	18.47	0.22	0.12	0.34
7	41.71	1.89	5.68	11.48	17.2	3.65	13.21	16.86	0.20	0.15	0.35
8	145.63	2.49	n.d.	13.30	13.3	n.d.	12.49	12.49	0.05	0.22	0.28
	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]
3	100	42.21	69.11	42.76	111.88	39.00	46.16	85.15	0.57	0.38	0.95
4	100	39.76	48.38	18.91	67.29	26.36	19.57	45.93	0.37	0.21	0.59
5											
6	100	8.92	9.38	8.63	18.01	6.83	10.96	17.79	0.21	0.12	0.33
7	100	4.53	13.61	27.52	41.14	8.76	31.66	40.42	0.48	0.36	0.84
8	100	1.71		9.13	9.13		8.58	8.58	0.04	0.15	0.19

BPA mass fluxes WWTP 1 (cont.)

week	mass flux								
	aer. sampling liquid	aer. sampling solid	aerr. sampling total	denitr. effluent liquid	denitr. effluent solid	denitr. effluent total	aer. effluent liquid	aer. effluent solid	aer. effluent total
	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]
3	0.53	0.33	0.86	202.72	135.87	338.58	85.57	52.95	138.52
4	0.35	0.14	0.49	156.80	89.66	246.46	61.17	23.92	85.08
5	n.m.	n.m.		n.m.	n.m.		n.m.	n.m.	
6	0.07	0.06	0.13	74.42	41.28	115.70	12.37	11.38	23.75
7	0.04	0.07	0.11	66.46	49.48	115.94	6.53	13.21	19.74
8	n.d.	0.08	0.08	18.32	74.53	92.84	n.d.	14.98	14.98
	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]
3	0.46	0.29	0.75	176.72	118.44	295.16	74.60	46.16	120.76
4	0.29	0.11	0.40	128.32	73.38	201.70	50.06	19.57	69.63
5									
6	0.06	0.06	0.12	71.68	39.76	111.45	11.92	10.96	22.88
7	0.09	0.18	0.27	159.34	118.61	277.95	15.66	31.66	47.32
8		0.06	0.06	12.58	51.17	63.75		10.28	10.28

BPA mass fluxes WWTP 1 (cont.)

week	mass flux			balance			
	separator liquid (calcul.)	separator solid (calcul.)	effluent separator (calcul.)	denitr.	aer.	separator	total
	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]
3	40.84	n.m.	40.84	-1.1	-70.9	7.6	-64.3
4	28.96	n.m.	28.96	-13.4	-78.7	19.6	-72.4
5	n.m.	n.m.					
6	5.28	n.m.	5.28	-24.9	-73.1	4.0	-94.1
7	2.88	n.m.	2.88	40.6	-78.9	-1.0	-39.4
8	n.d.	n.m.	n.d.	-78.3	-64.5	2.5	-142.8
	[%]	[%]	[%]	[%]	[%]	[%]	[%]
3	35.60		35.60	-0.9	-61.8	6.6	-56.1
4	23.70		23.70	-10.9	-64.4	16.1	-59.3
5							
6	5.09		5.09	-24.0	-70.4	3.8	-90.6
7	6.90		6.90	97.2	-189.2	-2.4	-94.4
8				-53.8	-44.3	1.7	-98.0

Table 20: BPA mass fluxes WWTP 2

week	mass flux										
	influent	effluent	denitr. recircul. liquid	denitr. recircul. solid	denitr. recircul. total	intern recirc. liquid	intern recirc. solid	intern recirc. total	denitr. sampling g liquid	denitr. sampling g solid	denitr. sampling total
	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]
3	178.3	47.2	64.6	34.2	98.7	28.1	30.5	58.6	0.63	0.41	1.04
4	178.3	36.2	13.1	9.7	22.7	7.5	10.2	17.7	0.27	0.18	0.45
5	112.4	14.6	15.9	10.7	26.7	9.1	11.2	20.4	0.22	0.21	0.43
6	110.5	6.5	8.1	8.4	16.6	4.8	8.9	13.7	0.18	0.13	0.31
7	48.9	2.7	6.0	5.3	11.3	3.5	5.7	9.2	0.16	0.15	0.31
8	142.2	3.2	n.d.	15.8	15.8	n.d.	13.5	13.5	0.03	0.18	0.22
	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]
3	100	26.5	36.2	19.1	55.3	15.7	17.1	32.9	0.35	0.23	0.58
4	100	20.3	7.3	5.4	12.8	4.2	5.7	9.9	0.15	0.10	0.25
5	100	13.0	14.2	9.5	23.7	8.1	10.0	18.1	0.20	0.18	0.38
6	100	5.9	7.3	7.6	15.0	4.3	8.1	12.4	0.16	0.12	0.28
7	100	5.4	12.2	10.9	23.1	7.2	11.6	18.8	0.33	0.30	0.63
8	100	2.3		11.1	11.1		9.5	9.5	0.02	0.13	0.15

BPA mass fluxes WWTP 2 (cont.)

week	mass flux								
	aer. sampling liquid	aer. sampling solid	aerr. sampling total	denitr. effluent liquid	denitr. effluent solid	denitr. effluent total	aer. effluent liquid	aer. effluent solid	aer. effluent total
	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]
3	0.39	0.21	0.60	197.8	126.9	324.7	57.7	30.5	88.2
4	0.08	0.06	0.14	92.3	62.0	154.2	13.8	10.2	24.0
5	0.10	0.06	0.16	75.8	70.0	145.9	16.8	11.2	28.0
6	0.05	0.05	0.10	62.0	44.4	106.4	8.6	8.9	17.6
7	0.04	0.03	0.07	55.5	50.7	106.2	6.3	5.7	12.0
8	n.d.	0.09	0.09	12.0	63.0	75.0	n.d.	16.7	16.7
	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]
3	0.22	0.12	0.34	110.9	71.1	182.0	32.3	17.1	49.4
4	0.04	0.03	0.08	51.8	34.8	86.5	7.7	5.7	13.4
5	0.09	0.06	0.14	67.5	62.3	129.8	14.9	10.0	24.9
6	0.04	0.05	0.09	56.0	40.2	96.2	7.8	8.1	15.9
7	0.07	0.07	0.14	113.6	103.8	217.4	12.9	11.6	24.5
8		0.07	0.07	8.4	44.3	52.7		11.8	11.8

BPA mass fluxes WWTP 2 (cont.)

week	mass flux			balance			
	separator liquid (calcul.)	separator solid (calcul.)	effluent separator (calcul.)	denitr.	aer.	separator	total
	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]	[µg/d]
3	29.6	n.m.	29.6	-9.9	-137.2	17.7	-129.5
4	6.3	n.m.	6.3	-64.0	-107.4	29.9	-141.5
5	7.6	n.m.	7.6	-13.2	-91.0	7.0	-97.2
6	3.8	n.m.	3.8	-34.2	-72.1	2.7	-103.6
7	2.8	n.m.	2.8	37.2	-82.9	-0.2	-45.8
8	n.d.	n.m.	n.d.	-96.4	-42.3	3.2	-138.7
	[%]	[%]	[%]	[%]	[%]	[%]	[%]
3	16.6		16.6	-5.6	-76.9	9.9	-72.6
4	3.5		3.5	-35.9	-60.2	16.8	-79.4
5	6.8		6.8	-11.7	-81.0	6.2	-86.5
6	3.5		3.5	-30.9	-65.3	2.4	-93.7
7	5.8		5.8	76.1	-169.6	-0.3	-93.8
8				-67.8	-29.7	2.3	-97.5

Table 21: Influent WWTP 1; corollary parameters 17 β -estradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-04-10	<0.03	0.51	27.3	156	405	7.89	580	5
2000-04-17	0.15	0.61	10.5	174	440	7.71	520	6
2000-04-24	<0.03	0.39	10.1	174	420	7.69	529	7
2000-05-02	<0.03	0.60	10.8	168	420	7.62	520	8
2000-05-08	0.13	0.21	13.9	194	370	7.695	529	9
2000-05-15	0.34	0.82	8.3	167	420	7.875	482	10
2000-05-22	0.11	0.63	8.0	155	390	7.82	472	11
2000-05-29	0.24	0.23	9.2	177	450	7.9	475	12
2000-06-05	0.23	0.42	13.2	170	410	7.38	538	13
2000-06-13	0.45	2.71	15.1	174	430	7.63	560	14
2000-06-19	<0.03	2.43	8.1	136	340	7.82	502	15

Table 22: Effluent WWTP 1; corollary parameters 17 β -estradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-04-10	0.24	15.8	0.36	4.8	20	7.07	458	5
2000-04-17	0.25	15.6	0.24	3.9	19	7.04	441	6
2000-04-24	<0.03	14.5	0.44	4.5	19	7.16	434	7
2000-05-02	0.27	20.1	0.13	4.6	17	6.73	477	8
2000-05-08	<0.03	16.1	0.17	3.5	13	7.43	427	9
2000-05-15	0.32	14.1	0.24	5.1	17	7.11	472	10
2000-05-22	0.43	16.0	0.47	8.4	29	7.00	479	11
2000-05-29	1.10	8.87	25.8	6.2	25	7.76	636	12
2000-06-05	2.10	8.74	2.06	11.8	46	7.27	483	13
2000-06-13	5.33	7.70	21.5	7.8	34	7.90	660	14
2000-06-19	8.89	7.16	2.90	5.4	28	7.38	543	15

Table 23: Denitrification vessel WWTP 1; corollary parameters 17 β -estradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-04-10	<0.03	0.68	14.8	11.6	41	3455	7.31	44	5
2000-04-17	0.09	1.10	9.38	8.7	34	3856	7.31	48	6
2000-04-24	<0.03	0.86	11.4	10.0	36	4457	7.38	31	7
2000-05-02	0.23	1.70	11.6	10.9	45	4490	7.23	51	8
2000-05-08	<0.03	0.85	9.24	6.6	27	5014	7.28	39	9
2000-05-15	<0.03	0.53	10.9	11.1	34	4445	7.21	58	10
2000-05-22	<0.03	0.72	10.1	15.3	49	3870	7.21	56	11
2000-05-29	0.20	0.53	27.7	17.9	51	2966	7.46	50	12
2000-06-05	0.30	0.62	12.0	16.6	56	2840	7.33	75	13
2000-06-13	0.40	0.79	29.5	18.3	55	2982	7.33	82	14
2000-06-19	0.30	0.95	12.3	12.2	35	2806	6.95	59	15

Table 24: Aeration vessel WWTP 1; corollary parameters 17 β -estradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-04-10	0.29	11.0	3.04	6.7	28	2913	6.79	20	5
2000-04-17	0.72	11.6	1.71	5.4	24	3180	6.72	26	6
2000-04-24	0.51	13.2	1.73	6.0	22	3664	6.77	13	7
2000-05-02	1.21	14.1	2.66	6.2	23	4129	6.60	32	8
2000-05-08	0.69	11.5	2.22	5.4	18	3693	7.06	22	9
2000-05-15	0.78	10.0	2.33	6.8	22	3903	6.86	45	10
2000-05-22	0.68	13.1	2.29	12.2	35	3078	6.73	49	11
2000-05-29	1.17	6.73	24.5	9.6	33	2468	7.36	25	12
2000-06-05	2.16	8.63	3.00	12.0	50	2356	6.83	41	13
2000-06-13	3.87	4.67	22.3	12.0	46	2516	7.33	45	14
2000-06-19	7.76	5.01	4.36	8.3	37	2362	7.53	38	15

Table 25: Influent WWTP 2; corollary parameters 17 β -estradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-04-10	0.16	0.30	38.8	217	555	7.82	730	5
2000-04-17	<0.04	0.32	16.0	175	430	7.83	543	6
2000-04-24	<0.04	0.34	16.2	194	470	7.44	535	7
2000-05-02	0.33	0.38	14.8	235	590	7.47	609	8
2000-05-08	<0.04	0.19	19.6	204	390	7.57	593	9
2000-05-15	0.21	0.61	9.2	179	470	7.82	492	10
2000-05-22	0.13	0.47	10.0	202	400	7.62	506	11
2000-05-29	0.35	0.45	13.6	224	550	7.68	543	12
2000-06-05	0.25	0.44	13.8	175	410	7.36	520	13
2000-06-13	0.51	2.08	15.4	196	460	7.63	580	14
2000-06-19	<0.04	2.66	7.5	161	390	7.76	525	15

Table 26: Effluent WWTP 2; corollary parameters 17 β -estradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-04-10	0.35	13.3	0.44	5.3	26	7.35	556	5
2000-04-17	0.19	14.5	0.22	4.0	15	7.18	443	6
2000-04-24	<0.03	14.4	0.18	4.9	20	7.26	445	7
2000-05-02	0.26	19.1	0.10	4.3	18	6.96	488	8
2000-05-08	0.15	16.2	0.10	4.3	18	7.17	464	9
2000-05-15	0.17	15.7	0.16	4.4	13	7.13	476	10
2000-05-22	0.25	14.8	0.19	4.5	18	7.07	465	11
2000-05-29	0.18	19.4	0.32	5.6	23	6.98	506	12
2000-06-05	0.16	15.7	0.42	5.7	23	7.07	473	13
2000-06-13	0.40	19.6	0.60	7.3	28	7.15	548	14
2000-06-19	0.36	17.6	0.27	5.7	22	7.16	543	15

Table 27: Denitrification vessel WWTP 2; corollary parameters 17 β -estradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	Woche
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-04-10	<0.03	1.31	11.4	10.0	38	3464	7.40	34	5
2000-04-17	0.14	2.21	6.64	8.2	30	3089	7.36	41	6
2000-04-24	<0.03	1.70	11.6	10.8	35	3618	7.30	39	7
2000-05-02	0.31	1.64	10.8	10.1	30	4313	7.22	341	8
2000-05-08	0.15	1.96	7.4	8.3	30	4528	7.27	52	9
2000-05-15	0.19	1.65	11.5	10.2	29	4555	7.15	58	10
2000-05-22	0.25	1.41	11.0	14.8	30	4367	7.19	57	11
2000-05-29	0.23	0.75	18.6	15.5	44	3796	7.20	83	12
2000-06-05	n.m.	1.13	11.5	12.8	44	4098	7.05	68	13
2000-06-13	0.76	1.13	14.3	10.0	36	3724	7.07	73	14
2000-06-19	1.17	2.20	12.2	11.9	31	4184	7.20	61	15

Table 28: Aeration vessel WWTP 2; corollary parameters 17 β -estradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-04-10	0.29	9.33	2.83	6.7	32	3154	6.84	20	5
2000-04-17	0.36	11.3	1.45	5.4	20	2357	6.89	25	6
2000-04-24	0.70	11.4	1.87	6.3	21	2747	6.88	16	7
2000-05-02	0.87	13.7	2.70	6.5	27	3671	6.83	28	8
2000-05-08	1.00	11.2	1.81	6.1	23	3796	6.92	27	9
2000-05-15	0.98	11.5	2.06	5.9	22	3858	6.86	34	10
2000-05-22	0.80	11.0	2.34	8.4	33	3869	6.80	36	11
2000-05-29	1.49	12.4	4.53	8.0	12	3308	6.78	44	12
2000-06-05	0.85	10.1	3.10	8.7	35	3458	6.80	38	13
2000-06-13	0.88	9.66	4.62	12.3	36	3458	6.86	49	14
2000-06-19	1.25	12.7	2.05	7.9	31	3092	6.88	42	15

Table 29: pH-value and temperature; 17 β -estradiol degradation experiments

pH-value		
n = 18	WWTP 1	WWTP 2
influent	7.74 (± 0.08 ; s = 0.16)	7.64 (± 0.09 ; s = 0.19)
denitrification	7.27 (± 0.05 ; s = 0.12)	7.23 (± 0.05 ; s = 0.11)
aeration (nitrification)	6.89 (± 0.14 ; s = 0.30)	6.85 (± 0.03 ; s = 0.06)
effluent	7.19 (± 0.16 ; s = 0.34)	7.14 (± 0.09 ; s = 0.20)
Temperature [°C]		
n = 53	WWTP 1	WWTP 2
aeration (nitrification)	23.8 (± 0.50 ; s = 1.8)	23.6 (± 0.50 ; s = 1.9)

Table 30: Debit hydraulic detention times; 17 β -estradiol degradation experiments

week	hydraulic detention time WWTP 1 and 2			
	denitr.	aerat.	separator	total
	[h]	[h]	[h]	[h]
5 - 15	1.46	1.46	1.9	16.0

Table 31: Sludge age WWTP 1 and 2; 17 β -estradiol degradation experiments

week	WWTP 1		WWTP 2	
	denitr.	aer.	denitr.	aer.
	[d]	[d]	[d]	[d]
5	28	28	28	28
6	28	28	28	28
7	35	35	35	35
8	26	26	26	26
9	21	21	21	21
10	21	21	21	21
11	21	21	21	21
12	84	25	84	22
13	84	20	168	17
14	168	105	252	84
15	420	420	420	420

Table 32: 17 β -estradiol concentrations WWTP 1

week	influent	denitr. liquid	denitr. solid	denitr. total	aer. liquid	aer. solid	aer. total	effluent
	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]
6	0.72	<0.0061	n.d.		n.q.	n.d.		<0.0084
7	0.75	0.051	n.d.		n.q.	n.d.		n.q.
8	0.89	0.048	n.q.		n.d.	n.d.		<0.0076
9	0.54	0.056	<11.1	<0.11	n.d.	n.d.		n.q.
10	1.81	0.066	n.q.		<0.0070	n.d.		0.030
11	3.25	n.m.	n.m.		0.022	<9.84	<0.052	0.057
12	3.45	n.m.	n.m.		0.026	n.q.		0.13

Table 33: Estrone concentrations WWTP 1

week	Influent	denitr. liquid	denitr. solid	denitr. total	aer. liquid	aer. solid	aer. total	effluent
	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]
6	0.0164	0.047	8.09	0.079	<0.0043	n.d.		n.q.
7	0.0085	0.16	4.48	0.18	0.0165	n.d.		n.d.
8	0.0105	0.31	19.38	0.39	0.0137	n.q.		n.q.
9	0.0103	0.23	14.45	0.31	0.021	n.d.		n.q.
10	0.021	0.29	15.72	0.36	0.043	n.q.		0.0112
11	0.030	n.m.	n.m.		0.0138	<2.85	<0.023	0.021
12	0.022	n.m.	n.m.		0.052	<10.2	<0.077	0.0127

Table 34: Estriol concentrations WWTP 1

WWTP 1	influent	denitr. liquid	denitr. solid	denitr. total	aer. liquid	aer. solid	aer. total	effluent
	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]
6	n.d.	n.d.	n.d.		n.d.	n.d.		n.d.
7	n.d.	0.06	n.d.		n.d.	n.d.		n.d.
8	n.q.	n.d.	n.d.		n.d.	n.d.		n.d.
9	n.d.	n.d.	n.d.		n.d.	n.d.		n.d.
10	n.d.	n.d.	n.d.		n.d.	19.73		n.d.
11	n.d.	n.m.	n.m.		n.d.	22.38		n.d.
12	n.d.	n.m.	n.m.		n.d.	21.89		n.d.

Table 35: 17 β -estradiol concentrations WWTP 2

week	influent	denitr.liquid	denitr.solid	denitr.total	aer.liquid	aer.solid	aer.total	effluent
	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kgDM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kgDM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]
6	0.82	0.018	n.d.		n.d.	n.d.		<0.027
7	0.80	0.015	n.d.		n.d.	n.d.		<0.0061
8	0.93	<0.047	<9.86	<0.09	0.010	<11.9	<0.054	0.012
9	0.59	0.059	n.q.		n.d.	n.d.		0.59
10	1.73	0.053	<5.89	<0.08	0.029	n.d.		0.028
11	3.89	n.m.	n.m.		0.0152	n.d.		0.041
12	3.34	n.m.	n.m.		<0.0068	n.q.		0.030

Table 36: Estrone concentrations WWTP 2

week	influent	denitr. liquid	denitr. solid	denitr. total	aer. liquid	aer. solid	aer. total	effluent
	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]
6	0.18	0.055	5.85	0.056	n.q.	n.d.		<0.0085
7	n.q.	0.053	5.96	0.054	0.0094	n.d.		n.q.
8	0.0157	0.22	<16.1	<0.23	0.024	<16.2	<0.084	n.q.
9	0.0089	0.26	12.98	0.26	0.021	n.q.		0.0089
10	0.0162	0.20	14.41	0.21	0.025	n.q.		<0.0042
11	0.031	n.m.	n.m.		0.13	17.03	0.194	0.019
12	0.018	n.m.	n.m.		0.036	7.10	0.059	0.0121

Table 37: Estriol concentrations WWTP 2

WWTP 2	influent	denitr. liquid	denitr. solid	denitr. total	aer. liquid	aer. solid	aer. total	effluent
	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]
6	n.d.	n.d.	n.d.		n.d.	n.d.		n.d.
7	n.d.	n.d.	n.d.		n.d.	n.d.		n.d.
8	n.d.	n.d.	n.d.		n.d.	n.d.		n.d.
9	n.d.	n.d.	n.d.		n.d.	n.d.		n.d.
10	n.d.	n.d.	n.d.		n.d.	15.91		n.d.
11	n.d.	n.m.	n.m.		n.d.	n.d.		n.d.
12	n.d.	n.m.	n.m.		n.d.	n.d.		n.d.

mass fluxes WWTP 2, week 8; 17 β -estradiol degradation experiment (cont.)

WWTP 2, week 8	aer. sampling liquid	aer. sampling solid	aer. sampling total	denitr. effluent liquid	denitr. effluent solid	denitr. effluent total	aer. effluent liquid	aer. effluent solid	aer. effluent total
mass flux E2 [μ g/d]	0.001	0.005	0.006	2.3	2.1	4.4	0.26	1.1	1.4
mass flux E1 [μ g/d]	0.003	0.007	0.01	10.9	3.4	14.3	0.61	1.5	2.1
mass flux E2 [nmol/d]	0.004	0.02	0.02	8.5	7.7	16.2	0.97	4.0	5.0
mass flux E1 [nmol/d]	0.01	0.03	0.04	40.5	12.6	53.1	2.24	5.6	7.8
mass flux E2+E1 [nmol/d]	0.01	0.04	0.06	49.0	23.0	69.2	3.21	9.6	12.8
mass flux in relation to system influent AD									
[%]	0.01	0.04	0.06	20.8	18.8	39.5	2.4	9.8	12.2
[%]	1.5	3.6	5.1	5,804	1,808	7,612	322	797	1,119
[%]	0.03	0.10	0.14	118	48.7	166	7.7	23.0	30.7
mass flux in relation to sum E1+E2 mass flux									
[%]	30.1	42.0	39.0	17.4	37.9	23.4	30.1	42.0	39.0
[%]	69.9	58.0	61.0	82.6	62.1	76.6	69.9	58.0	61.0
[%]	100	100	100	100	100	100	100	100	100

mass fluxes WWTP 2, week 8; 17 β -estradiol degradation experiment (cont.)

WWTP 2, week 8	separator liquid (calcul.)	separator solid (calcul.)	effluent separator (calcul.)	denitr.	aer.	separator	total
mass flux E2 [μ g/d]	0.13		0.13	-9.3	-1.8	0.02	-11.0
mass flux E1 [μ g/d]	0.29		0.3	10.4	-10.2	-0.3	-0.11
mass flux E2 [nmol/d]	0.46		0.5	-34.0	-6.4	0.1	-40.4
mass flux E1 [nmol/d]	1.1		1.1	38.3	-37.8	-0.9	-0.4
mass flux E2+E1 [nmol/d]	1.5		1.5	4.3	-44.2	-0.9	-40.8
mass flux in relation to system influent AD							
[%]	1.1		1.1	-83.0	-15.7	0.2	-98.6
[%]	153		153	5,497	-5,421	-136	-59.8
[%]	3.7		3.7	10.3	-106	-2.1	-97.9
mass flux in relation to sum E1+E2 mass flux							
[%]	30.1		30.1		14.5	-7.0	99.0
[%]	69.9		69.9		85.5	107	1.0
[%]	100		100	100	100	100	100

Table 40: Influent WWTP 1; 17 α -ethinylestradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-06-26	0.19	0.45	7.36	180	430	7.89	506	1
2000-07-03	0.29	0.11	10.0	160	380	7.61	542	2
2000-07-10	0.03	0.29	10.6	248	620	7.71	383	3
2000-07-18	0.21	0.11	6.92	189	430	7.74	480	4
2000-07-24	0.20	0.14	6.91	160	390	7.73	474	5
2000-08-02	0.03	0.08	17.0	149	370	7.60	549	6
2000-08-08	0.22	0.15	6.91	155	330	7.81	419	7
2000-08-14	0.19	0.14	11.2	126	290	6.98	408	8
2000-08-21	0.11	n.m.	7.18	138	290	7.67	488	9
2000-08-28	n.m.	0.40	15.4	129	270	7.96	458	10

Table 41: Effluent WWTP 1; 17 α -ethinylestradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-06-26	0.67	15.5	0.18	15.6	45	7.01	48	1
2000-07-03	0.64	17.7	0.20	7.2	32	6.93	471	2
2000-07-10	1.07	17.7	0.22	13.5	50	6.99	471	3
2000-07-18	0.24	18.9	0.24	5.4	18	7.08	464	4
2000-07-24	0.51	15.6	0.10	5.9	22	7.15	466	5
2000-08-02	0.69	14.4	0.31	5.8	34	7.67	425	6
2000-08-08	0.50	13.7	0.30	5.0	26	7.08	429	7
2000-08-14	0.54	16.9	0.26	5.3	26	7.03	442	8
2000-08-21	1.73	14.9	0.26	5.3	32	7.26	474	9
2000-08-28	0.65	15.2	0.12	5.1	21	7.42	447	10

Table 42: Denitrification vessel WWTP 1; 17 α -ethinylestradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/L]		[μ s/cm]	
2000-06-26	0.03	0.46	9.17	30.3	81	3082	7.37	529	1
2000-07-03	0.25	0.28	12.5	22.5	74	3492	7.30	576	2
2000-07-10	0.03	0.70	10.6	19.1	56	3914	7.31	543	3
2000-07-18	0.16	0.46	9.47	14.2	43	3920	7.24	513	4
2000-07-24	n.m.	0.49	10.6	15.2	47	4130	7.08	568	5
2000-08-02	0.24	0.12	9.89	14.0	54	3048	7.16	482	6
2000-08-08	0.16	0.25	7.79	14.6	47	3480	7.21	471	7
2000-08-14	0.41	0.60	7.65	9.6	37	3106	6.64	483	8
2000-08-21	0.31	0.73	8.15	11.9	49	4252	7.01	555	9
2000-08-28	0.49	1.07	7.63	10.5	34	4264	7.27	510	10

Table 43: Aeration vessel WWTP 1; 17 α -ethinylestradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/L]		[μ s/cm]	
2000-06-26	0.31	12.1	1.57	18.9	54	2786	6.78	481	1
2000-07-03	0.47	10.7	2.02	11.1	38	2928	6.74	477	2
2000-07-10	0.46	12.5	2.68	10.7	38	2570	6.86	474	3
2000-07-18	0.16	13.6	1.63	8.3	29	3582	6.83	465	4
2000-07-24	0.35	8.69	2.39	9.2	29	3628	6.98	478	5
2000-08-02	0.50	11.4	1.37	8.0	35	3280	6.93	424	6
2000-08-08	0.44	11.5	1.50	8.0	28	3608	6.83	421	7
2000-08-14	0.59	12.7	1.64	8.4	38	2832	6.35	445	8
2000-08-21	0.68	11.9	1.29	7.8	40	3728	7.07	466	9
2000-08-28	0.72	15.5	0.45	7.6	31	4813	7.18	457	10

Table 44: Influent WWTP 2; 17 α -ethinylestradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
26.6.00 7:00	0.21	0.53	10.73	177	410	7.93	525	
3.7.00 7:00	0.38	0.05	13.22	167	400	7.39	551	
10.7.00 12:15	0.03	1.76	8.98	181	470	7.66	459	
2000-07-18	0.21	0.03	6.67	167	420	7.70	474	1
2000-07-24	0.03	0.35	7.43	163	390	7.75	485	2
2000-08-02	0.03	0.02	20.3	144	370	7.67	564	3
2000-08-08	0.03	0.11	8.49	139	380	7.65	436	4
2000-08-14	0.40	0.21	13.2	118	250	6.98	480	5
2000-08-21	0.85	n.m.	7.33	130	270	7.63	512	6
2000-08-28	n.m.	0.67	11.11	114	250	8.02	436	7

Table 45: Effluent WWTP 2; 17 α -ethinylestradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μ s/cm]	
2000-06-26	0.99	13.6	0.52	17.4	49	7.18	486	1
2000-07-03	0.57	19.4	0.22	6.8	28	6.94	491	2
2000-07-10	0.43	18.7	0.26	7.2	28	6.88	471	3
2000-07-18	0.32	18.8	0.73	6.9	23	6.94	478	4
2000-07-24	0.49	16.5	0.26	6.1	22	7.14	474	5
2000-08-02	0.75	10.6	0.84	5.7	26	7.17	425	6
2000-08-08	0.60	13.3	0.45	5.4	26	7.25	419	7
2000-08-14	0.72	15.1	0.30	6.0	25	6.35	441	8
2000-08-21	1.39	14.5	0.24	6.5	33	7.35	473	9
2000-08-28	0.81	12.7	0.30	5.6	24	7.17	444	10

Table 46: Denitrification vessel WWTP 2; 17 α -ethinylestradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/L]		[μ s/cm]	
2000-06-26	0.18	2.11	8.22	25.8	72	2236	7.41	541	1
2000-07-03	0.32	0.97	13.1	22.6	66	3374	7.17	481	2
2000-07-10	0.03	1.45	19.0	23.6	65	4186	7.22	563	3
2000-07-18	0.21	1.62	11.2	17.2	49	3790	7.18	568	4
2000-07-24	0.29	1.02	9.71	15.9	45	3466	7.10	561	5
2000-08-02	0.35	1.52	8.70	12.3	49	2944	7.19	492	6
2000-08-08	0.21	1.21	7.61	12.1	30	2924	7.24	474	7
2000-08-14	0.37	1.50	8.65	12.5	42	2954	6.58	504	8
2000-08-21	n.m.	1.53	8.30	12.1	45	3180	7.29	542	9
2000-08-28	n.m.	0.44	11.64	11.1	41	3752	7.37	507	10

Table 47: Aeration vessel WWTP 2; 17 α -ethinylestradiol degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/L]		[μ s/cm]	
2000-06-26	0.51	10.9	0.59	19.0	53	1922	6.88	486	1
2000-07-03	0.20	9.85	2.93	10.5	37	3208	6.76	589	2
2000-07-10	0.03	12.4	2.96	10.3	33	3580	6.86	474	3
2000-07-18	0.03	14.0	2.07	9.1	28	3482	6.64	471	4
2000-07-24	0.29	10.2	2.43	8.8	30	2914	6.96	511	5
2000-08-02	0.64	11.5	1.52	7.4	37	2054	6.87	439	6
2000-08-08	0.68	12.1	1.43	7.4	29	2634	6.88	419	7
2000-08-14	0.78	12.7	1.66	8.9	34	2572	6.16	448	8
2000-08-21	2.16	13.4	0.90	8.8	41	3184	7.01	476	9
2000-08-28	1.22	9.75	1.10	7.8	30	3722	6.94	453	10

Table 48: pH-value and temperature; 17 α -ethinylestradiol degradation experiments

pH-value		
n = 10	WWTP 1	WWTP 2
influent	7.67 (± 0.17 ; s = 0.27)	7.64 (± 0.18 ; s = 0.29)
denitrification	7.16 (± 0.13 ; s = 0.21)	7.18 (± 0.14 ; s = 0.23)
aeration (nitrification)	6.86 (± 0.14 ; s = 0.22)	6.80 (± 0.15 ; s = 0.25)
effluent	7.16 (± 0.14 ; s = 0.23)	7.04 (± 0.18 ; s = 0.28)
temperature [°C]		
n = 50	WWTP 1	WWTP 2
aeration (nitrification)	22.2 (± 0.59 ; s = 2.1)	22.3 (± 0.60 ; s = 2.2)

Table 49: Hydraulic detention time WWTP 1 and 2; 17 α -ethinylestradiol degradation experiments

week	hydraulic detention time WWTP 1				hydraulic detention time WWTP 2			
	denitr.	aerat.	separator	total	denitr.	aerat.	separator	total
	[h]	[h]	[h]	[h]	[h]	[h]	[h]	[h]
3	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
4	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
5	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
6	1.44	1.44	1.86	14.07	1.41	1.41	1.80	13.77
7	1.48	1.48	1.91	14.50	1.45	1.45	1.79	13.61
8	1.43	1.43	1.81	14.21	1.38	1.38	1.65	13.30
9	1.46	1.46	1.84	13.94	1.39	1.39	1.66	13.06
10	1.53	1.53	1.99	14.30	1.39	1.39	1.63	13.32

Table 50: Sludge age WWTP 1 and 2; 17 α -ethinylestradiol degradation experiments

week	WWTP 1		WWTP 2	
	denitr.	aer.	denitr.	aer.
	[d]	[d]	[d]	[d]
1	420	420	420	420
2	21	21	21	21
3	20	20	20	20
4	21	21	21	21
5	21	21	21	21
6	21	21	21	21
7	21	21	21	21
8	20	20	20	20
9	21	21	21	21
10	21	21	21	21

Table 51: 17 α -ethinylestradiol concentration WWTP 1

week	influent	denitr. liquid	denitr. solid	denitr. total	aer. liquid	aer. solid	aer. total	effluent
	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]
3	2.68	n.q.	76.7		n.q.	n.d.		n.m.
4	n.m.	1.20	511	1.20	1.34	399	1.34	n.m.
5	n.d.	n.m.	200		n.m.	374		n.q.
6	n.m.	n.m.	n.m.		n.m.	n.m.		n.m.
7	n.m.	0.83	304	0.83	<0.38	262	<1.33	n.m.
8	n.d.	<0.29	233		<1.08	625	<3.13	n.d.
9	n.q.	n.q.	631		n.d.	581		n.d.
10	n.d.	n.m.	n.m.		n.m.	n.m.		n.d.

Table 52: 17 α -ethinylestradiol concentration WWTP 2

week	influent	denitr. liquid	denitr. solid	denitr. total	aer. liquid	aer. solid	aer. total	effluent
	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]	[$\mu\text{g/kg DM}$]	[$\mu\text{g/l}$]	[$\mu\text{g/l}$]
3	1.86	<0.31	122	<0.82	n.q.	n.d.		n.m.
4	n.m.	1.34	406	2.88	0.89	243	1.60	n.m.
5	1.68	n.m.	269		n.m.	369		n.q.
6	n.m.	n.m.	n.m.		n.m.	n.m.		n.m.
7	n.m.	n.d.	396		n.d.	610		n.m.
8	n.d.	<0.77	170	<1.27	<1.12	818	<2.80	n.d.
9	0.33	n.d.	506		n.d.	610		n.d.
10	0.94	n.m.	n.m.		n.m.	n.m.		n.d.

Table 53: Volume fluxes WWTP 1; 17 α -ethinylestradiol degradation experiment

week	influent table water	influent diet / analyte solution	influent total	denitr. recircul.	intern recircul.	effluent denitr.	effluent aer.	sampling denitr. / aer. resp.	effluent separator (calculated)	effluent (measured)
	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]	[ml/h]
3	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	6.3	n.m.	n.m.
4	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	6.0	n.m.	n.m.
5	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	6.0	n.m.	n.m.
6	514	54.6	568.6	1.001	509	2.073	1.065	6.0	557	560
7	500	51.8	551.8	984	496	2.026	1.036	6.0	540	544
8	511	52	563.0	988	544	2.089	1.095	6.3	550	557
9	511	62.7	573.7	969	511	2.047	1.072	6.0	562	601
10	503	56	559.3	960	447	1.960	994	6.0	547	517

17 α -ethinylestradiol mass flux WWTP 1 (cont.)

week	separator liquid (calcul.)	separator solid (calcul.)	effluent separator (calcul.)	denitr.	aer.	separator	total
	[$\mu\text{g/d}$]	[$\mu\text{g/d}$]	[$\mu\text{g/d}$]	[$\mu\text{g/d}$]	[$\mu\text{g/d}$]	[$\mu\text{g/d}$]	[$\mu\text{g/d}$]
3	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
4	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
5	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
6	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
7	4.93		4.93	n.m.	-36.9	n.m.	n.m.
8	14.21		14.21	-95.3	106.5	-10.7	0.6
9	3.59		3.59	14.8	-17.9	0.2	-2.8
10	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
	[%]	[%]	[%]	[%]	[%]	[%]	[%]
8	395		395	-2,650	2.963	-296	16.2
9	49.0		49.0	203	-244	3.4	-37.7
10							

Table 58: Effluent WWTP 1; NP_{1,2}EO degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μs/cm]	
2001-10-29	1.12	14.1	1.20	n.m.	n.m.	6.74	382	2
2001-11-05	0.45	14.3	1.14	15.9	63	6.78	408	3
2001-11-12	0.39	12.2	0.30	6.5	31	6.86	364	4
2001-11-19	0.38	15.1	0.28	10.5	39	6.64	401	5
2001-11-28	0.30	10.5	0.28	11.1	47	6.86	416	6
2001-12-03	0.17	10.5	0.37	14.0	37	7.26	363	7
2001-12-10	0.66	9.60	1.05	7.7	31	7.38	430	8
2001-12-17	0.13	2.25	22.1	25.6	38	7.62	481	9
2002-01-07	8.47	1.03	1.43	8.1	41	7.40	419	12
2002-01-14	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	13

Table 59: Denitrification vessel WWTP 1; NP_{1,2}EO degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/L]		[μs/cm]	
2001-10-29	0.12	1.20	11.3	n.m.	n.m.	3322	7.39	417	2
2001-11-05	0.1	1.08	10.5	21.1	69	3404	7.30	440	3
2001-11-12	0.11	0.23	9.10	16.8	62	3238	7.35	403	4
2001-11-19	0.18	0.73	8.30	20.6	61	3068	7.19	452	5
2001-11-28	0.13	1.09	8.57	12.7	40	2866	7.16	462	6
2001-12-03	0.12	1.16	7.62	32.4	n.m.	2810	7.34	418	7
2001-12-10	0.55	0.10	7.40	21.9	61	2872	7.41	490	8
2001-12-17	0.1	0.56	23.5	26.5	84	1486	7.74	531	9
2002-01-07	1.07	0.58	8.80	16.5	58	2030	7.49	486	12
2002-01-14	n.m.	n.m.	n.m.	n.m.	n.m.	1980	n.m.	n.m.	13

Table 62: Effluent WWTP 2; NP_{1,2}EO degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[µs/cm]	
2001-10-29	1.02	15.2	1.50	n.m.	n.m.	6.63	415	2
2001-11-05	0.46	16.0	0.79	9.0	38	6.55	413	3
2001-11-12	1.98	13.3	0.40	8.6	43	6.84	365	4
2001-11-19	0.47	14.3	0.44	9.3	30	6.88	429	5
2001-11-28	0.75	6.93	14.3	8.9	38	7.27	510	6
2001-12-03	0.34	10.5	0.25	9.2	24	7.04	372	7
2001-12-10	0.79	12.2	0.63	8.4	33	7.06	431	8
2001-12-17	1.04	11.7	1.31	8.8	38	7.1	379	9
2002-01-07	1.90	10.7	0.21	6.9	35	6.85	351	12
2002-01-14	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	13

Table 63: Denitrification vessel WWTP 2; NP_{1,2}EO degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[µs/cm]	
2001-10-29	0.14	1.70	10.7	n.m.	n.m.	3498	7.27	460	2
2001-11-05	0.09	2.14	11.4	23.0	76	3724	7.21	453	3
2001-11-12	0.11	1.68	8.50	13.1	46	3280	7.35	404	4
2001-11-19	1.04	2.88	8.03	14.2	61	3076	7.19	482	5
2001-11-28	0.97	6.94	12.3	14.9	41	2684	7.38	547	6
2001-12-03	0.14	1.15	7.76	15.9	34	3260	7.25	432	7
2001-12-10	0.68	1.63	8.17	12.3	39	3152	7.28	488	8
2001-12-17	0.64	1.23	10.3	14.0	45	3720	7.24	460	9
2002-01-07	0.38	1.74	7.04	11.4	39	2462	7.37	443	12
2002-01-14	n.m.	n.m.	n.m.	n.m.	n.m.	2326	n.m.	n.m.	13

Table 64: Aeration vessel WWTP 2; NP_{1,2}EO degradation experiment

date	NO ₂ -N	NO ₃ -N	NH ₄ -N	DOC	COD	DM	pH	cond.	week
	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]	[mg/l]		[μs/cm]	
2001-10-29	0.57	11.9	3.10	n.m.	n.m.	3070	6.69	420	2
2001-11-05	0.24	12.1	3.03	13.7	47	3314	6.58	418	3
2001-11-12	0.38	10.2	1.50	8.9	42	2974	6.80	369	4
2001-11-19	0.44	14.8	0.36	10.4	48	2770	6.44	439	5
2001-11-28	0.19	0.51	19.1	8.8	34	2492	7.04	511	6
2001-12-03	0.21	10.9	1.16	12.1	33	2384	6.66	387	7
2001-12-10	0.92	12.7	0.43	8.5	32	2880	6.71	439	8
2001-12-17	1.07	11.7	0.86	9.5	34	2096	6.86	385	9
2002-01-07	1.63	11.0	0.28	8.6	37	2304	6.78	384	12
2002-01-14	n.m.	n.m.	n.m.	n.m.	n.m.	2456	n.m.	n.m.	13

Table 65: pH-value and temperature WWTP 1 and 2; NP_{1,2}EO degradation experiments

pH-value		
n = 10	WWTP 1	WWTP 2
influent	7.76 (± 0.11; s = 0.16)	7.74 (± 0.03; s = 0.04)
denitrification	7.37 (± 0.11; s = 0.17)	7.28 (± 0.05; s = 0.07)
aeration (nitrification)	6.81 (± 0.20; s = 0.31)	6.73 (± 0.11; s = 0.17)
effluent	7.06 (± 0.23; s = 0.36)	6.91 (± 0.15; s = 0.23)
temperature [°C]		
	WWTP 1 (n = 58)	WWTP 2 (n = 56)
aeration (nitrification)	19.9 (± 0.27; s = 1.1)	19.9 (± 0.31; s = 1.2)

Table 66: Hydraulic detention time WWTP 1 and 2; NP_{1,2}EO degradation experiments

week	hydraulic detention time WWTP 1				hydraulic detention time WWTP 2			
	denitr.	aerat.	separator	total	denitr.	aerat.	separator	total
	[h]	[h]	[h]	[h]	[h]	[h]	[h]	[h]
2	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	15.6
3	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.
4	1.43	1.43	1.97	15.7	1.50	1.50	2.01	15.0
5	1.54	1.54	2.17	16.9	1.54	1.54	2.07	15.1
6	1.43	1.43	1.81	14.5	1.55	1.55	2.08	16.2
7	1.19	1.19	1.89	14.6	1.21	1.21	2.00	14.8
8	1.29	1.29	1.89	14.8	1.22	1.22	1.83	13.2

Table 67: Sludge age WWTP 1 and 2; NP_{1,2}EO NP_{1,2}EO degradation experiments

week	WWTP 1		WWTP 2	
	denitr.	aer.	denitr.	aer.
	[d]	[d]	[d]	[d]
3	20	20	20	20
4	20	20	20	20
5	20	20	20	20
6	20	20	20	20
7	20	20	420	420
8	20	20	20	20
9	20	20	20	20
12	20	20	20	20
13	20	20	20	20

Table 68: EDC concentrations determined 2000-05-26, full-scale WWTP no. 6

EDC	influent	effluent	excess sludge	dewatered sludge
	[µg/l]	[µg/l]	[µg/kg DM]	[µg/kg DM]
NP	0.78	1.06	1,240	1185
OP	0.40	0.37	294	347
BPA	1.27	0.36	150	202
EE2	n.c.	n.c.	1	10

Table 69: EDC concentrations determined 2001-11-27, full-scale WWTP no. 6

EDC	influent	denitr.	aer.	p-elim.	effluent
	[µg/l]	[µg/l]	[µg/l]	[µg/l]	[µg/l]
NP	n.d.	0.056	0.054	0.070	1.75
OP	n.d.	0.001	0.005	0.016	0.048
BPA	n.d.	0.553	0.162	0.282	1.10