

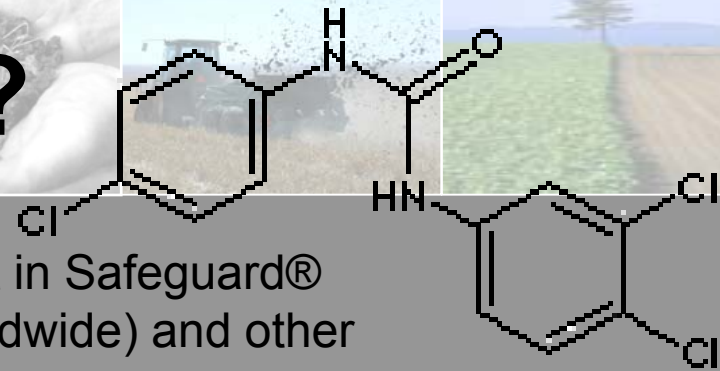


# **Fate and Transport of Biosolids-Borne Triclocarban**

**Funded by the USEPA  
In collaboration with the Procter & Gamble Company**

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# What is Triclocarban?



The antimicrobial active ingredient in Safeguard® (the No. 1 antibacterial soap worldwide) and other antibacterial bar soaps

- May also be added as a preservative to other personal care products



- Depending on concentration, may be bacteriostatic or bactericidal
- A membrane active compound
- Adsorbs to cell membranes, leading to interstitial protein function loss and/or membrane semipermeability loss
- Primarily inhibits growth of gram-positive bacteria (e.g. staphylococcus)
- High Production Volume chemical (HPV)  
227,000 – 454,000 kg TCC used/year in US (500,000 – 1,000,000 lbs)

# Environmental Fate and Toxicity Data

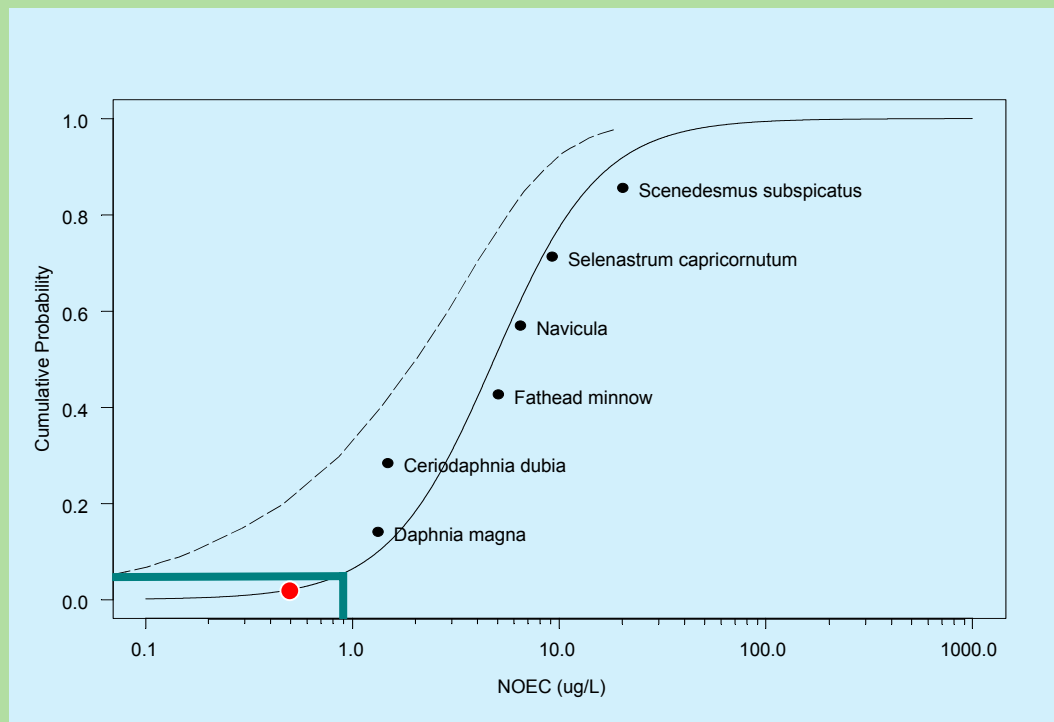
## Wastewater treatment removal

### McAvoy et al.

- 14.6 ppb – influent
- 0.54 ppb – effluent
- 96% removal

### Heidler et al.

- 6 ppb – influent
- 0.17 ppb – effluent
- 97% removal
  - 3% in effluent
  - 21% degraded/lost
  - 76% in dewatered sludge
  - 50mg/kg dry biosolids





# Biosolids-Borne TCC Data Needs

- Concentration of TCC in biosolids

- Effects of biosolids preparation methods on biosolids-borne TCC concentrations

- Fate of TCC in biosolids-amended soils

- Physicochemical properties of TCC

- Mobility of TCC in biosolids-amended soils

- Effects of biosolids-borne TCC on crops and soil microbial populations

- Assessment of human exposure routes and exposure estimates associated with TCC-contaminated biosolids



# Biosolids-Borne TCC Data Needs

- Concentration of TCC in biosolids

- Develop extraction and analytical methods
- Effects of biosolids preparation methods on biosolids-borne TCC concentrations
- Confirm or challenge reported biosolids-borne TCC concentrations

- Fate of TCC in biosolids-amended soils

- Identify biosolids treatment processes that may result in elevated TCC concentrations
- Physicochemical properties of TCC

- Conduct preliminary assessment of TCC biodegradation in biosolids-amended soils

- Measure  $K_{ow}$



# Extraction Procedure Development

## Round 1

- 6 biosolids
- Spiked with 0.0002mg 4-chlorophenyl-U-<sup>14</sup>C Triclocarban ( $10^5$  dpms) / gram dry weight biosolids
- Vortex 4 hours
- Equilibrate 20 hours
- Extraction (100% MeOH)
  - Freeze dry, ASE
    - $69 \pm 11\%$  recovery
  - Freeze dry, shake-flask
    - $66 \pm 7\%$  recovery
  - Refrigerate, shake-flask
    - $56 \pm 8\%$  recovery
  - Immediate shake-flask
    - $54 \pm 13\%$  recovery

## Round 2

- 3 biosolids
- Spiked with 0.0002mg 4-chlorophenyl-U-<sup>14</sup>C Triclocarban ( $10^5$  dpms) / gram dry weight biosolids
- Extraction
  - Freeze dry, ASE (100% acetone)
    - 1500 psi, 100°C
    - $82 \pm 4\%$  recovery
  - Freeze dry, shake-flask (50:50 MeOH/acetone)
    - $84\% \pm 8\%$  recovery



- Percent recoveries calculated by LSC



- Confirmation of parent compound by RAD-TLC

# Biosolids Extraction

## Biosolids Set 1

- Weigh 1 g biosolids (dry wt.)
- Freeze dry
- Extract
  - shake flask
  - 50:50 MeOH:acetone
- Dry extract and bring to volume
- d7-TCC internal standard
- Analyze by HPLC/MS

## Biosolids Set 2

- Weigh 1 g biosolids (dry wt.)
- d7-TCC spike (1ug)
- Equilibrate
- Freeze dry
- Extract
  - shake flask
  - 50:50 MeOH:acetone
- Dry extract and bring to volume
- $^{13}\text{C}$ -TCC internal standard
- Analyze by HPLC/MS



# Analytical Procedure



- Waters Alliance 2795 LC Mobile Phase
  - Solvent A
    - 25% 5:95 MeOH/H<sub>2</sub>O 0.01 AmAc
  - Solvent B
    - 75% 95:5 MeOH/H<sub>2</sub>O 0.01 AmAc
- Column
  - Luna c18 2x100x3mic
  - 40° C
- Injection volume
  - 3-4 uL
- ES- ionization mode
- Fragment masses of interest
  - 160 m/z
  - 287 m/z
  - 313 m/z
  - 289 m/z
  - 315 m/z
  - 293 m/z
  - 319 m/z
  - 295 m/z
  - 320 m/z

# TCC and TCS Concentrations in Biosolids

<u>Biosolids</u>	<u>Treatment Process</u>	<u>TCC Concentration (mg/kg)</u>	<u>TCS Concentration (mg/kg)</u>
DYMK	Mixed Compost	7 ± 2	
DYSK	Compost	5 ± 0.5	
GRBC	Aerobic Digestion	6 ± 1	
ORBC-BL	Untreated	21 ± 1	
ORBC-AL	Lime stabilization	15 ± 0.8	
GEPZ	Anaerobic digestion Pellets	24 ± 3	
UNKD	Anaerobic digestion	43 ± 3	40 + 3
UNKG	Anaerobic digestion	35 ± 1	31 + 0.6
CFBC	Anaerobic digestion (50 days)	34 ± 2	
UNKH	Anaerobic digestion	31 ± 0.7	25 + 1
UNKB	Anaerobic digestion	25 ± 1	33 + 0.7
UNKC	Anaerobic digestion (33 days)	24 ± 1	21 + 0.8
UNKF	Anaerobic digestion	23 ± 0.3	20 + 1
RCKF	Anaerobic digestion (30 days)	18 ± 3	
CHBC	Anaerobic digestion	14 ± 0.8	6
CLBC	Anaerobic digestion	13 ± 0.9	7 + 0.3
OSBC	Anaerobic digestion (26 days)	12 ± 2	
UNKE	Anaerobic digestion	10 ± 0.3	22 + 0.4
UNKI	Anaerobic digestion	8 ± 0.4	1 + 0.1
UNKJ	Unknown	31 ± 0.4	22 + 0.4
UNKK	Unknown	31 ± 1	23 + 1
UNKL	Unknown	12 ± 0.5	11 + 2



# TCC and TCS Concentrations in Biosolids

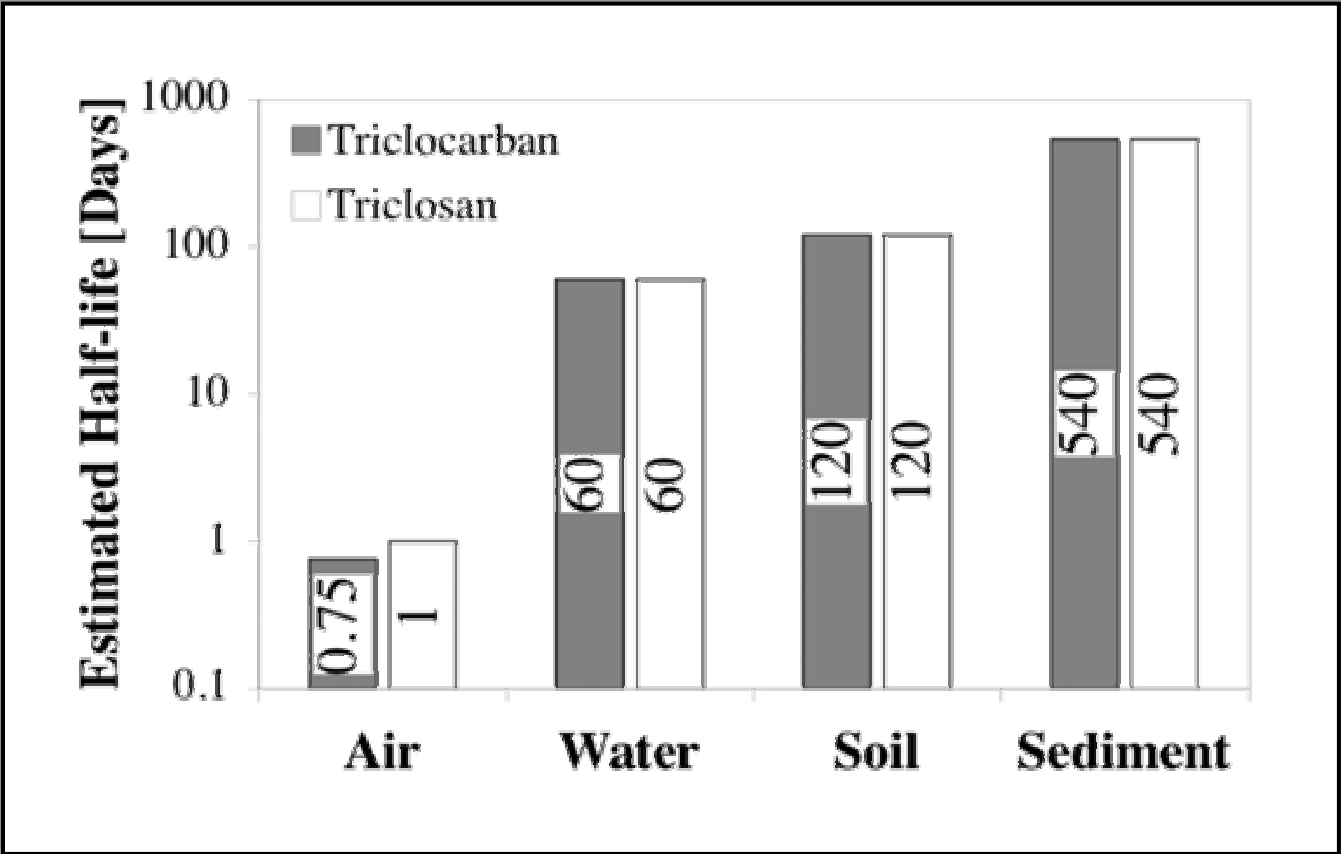
	<u>Present Study</u>	<u>Previous Publications</u>		
<u>TCC</u>				
<b>Average (mg/kg)</b>	22	51	40	
<b>Number of Treatment Plants</b>	13	1	10	
<b>Author</b>		(Heidler et al, 2006)	(Monsanto, 1981)	
<u>TCS</u>				
<b>Average (mg/kg)</b>	20	30	1.2	29
<b>Number of Treatment Plants</b>	10	1	20	1
<b>Author</b>		(Heidler et al, 2007)	(Bester, 2003)	(McAvoy, 2002)



# Environmental Persistence of TCC and TCS

Half-lives of triclocarban and triclosan in air, water, soil, and sediment, estimated using quantitative structure-activity relationship (QSAR) analysis

(Halden and Paull, 2005)



# Biodegradation Preliminary Study

## Sample Preparation

- 12 biotic
- 12 abiotic (20mL HgCl<sub>2</sub> / 30 g solids)
- 8 abiotic (10mL HgCl<sub>2</sub> / 30 g solids)
- 1:11 biosolids/soil (dry weight)
- 100,000 dpm per sample



## Procedure

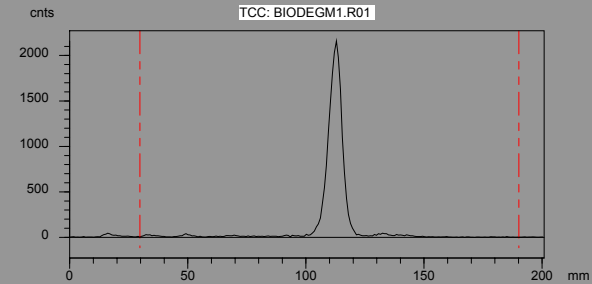
- Base traps removed at 1, 2, 5, 7, 17, and 38 days
- 4 samples removed at 7, 17, and 38 days
- Sequential extraction and mass balances calculated



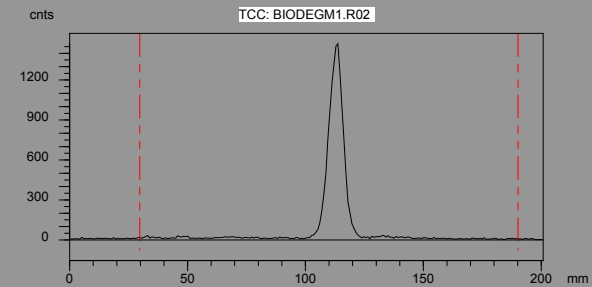
# Biodegradation Preliminary Study

<u>Fraction</u>	<u>% Recovered</u>					
	Day 7 Biotic (n=2)	Day 7 Abiotic (n=2)	Day 17 Biotic (n=2)	Day 17 Abiotic (n=2)	Day 38 Biotic (n=2)	Day 38 Abiotic (n=2)
H2O	0.95	0.68	0.83	0.98	0.59	0.16
MeOH	71	79	68	79	57	62
NaOH	1.8	1.3	2.4	1.7	3.0	1.6
CO2	2.9	0.21	4.1	0.35	6.7	0.23
Combustion	9.3	7.6	12.0	7.6	18	10
Total	86	89	87	90	85	74

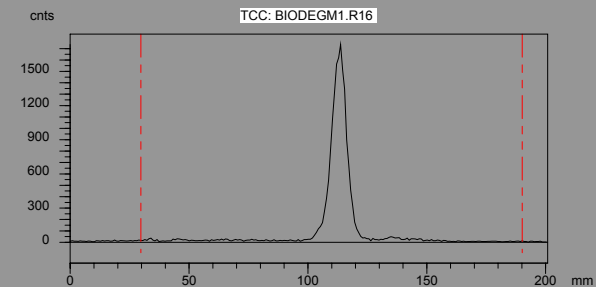
# Check for Biodegradation Intermediates



- Standard, with spike



- MeOH extract at 7 days



- MeOH extract at 17 days



# $K_{ow}$ Determination

- Previously measured  $\log K_{ow}$  values: 4.2, 5.8, 6
- Previously calculated  $\log K_{ow}$  values: 4.9
- OPPTS Guideline 830.7550 – Shake flask method

<b>n-octanol/water ratio</b>	<b><math>\log K_{ow}</math></b>
1:1	3.6
1:1	3.5
1:2	3.4
1:2	3.6
2:1	3.4
2:1	3.5
<b>Average</b>	<b><math>3.5 \pm 0.06</math></b>





# Remaining Data Needs

- TCC solubility confirmation
- Soil column leaching and plant uptake of TCC
- Measurement of biosolids sorption/desorption isotherms
- Longer term biosolids-amended soil TCC biodegradation
- Evaluation of TCC effects on soil microbial populations
- Assessment of human exposure routes and exposure estimates associated with TCC contaminated biosolids

