of the magnetic field because they occur too quickly. This particular magnetometer has its resonant frequency at $f_{\text{res}} \sim 10$ kHz when unloaded, which decreases to an estimated value of 5 kHz when loaded with the ~0.8-µg sample. Shown in Fig. 3b is the calibrated magnetization of the sample versus inverse magnetic field. The quantum oscillations are periodic in the inverse magnetic field. The quantum oscillation is particularly well suited for large samples and samples with rapid quantum oscillations. An alternate Si micromachined magnetometer design, the cantilever magnetometer (11), competes more directly with the trampoline magnetometer. This type of magnetometer uses a micromachined Si cantilever beam that holds the sample. The reported noise values are comparable to those for our trampoline magnetometer, although to date the cantilever magnetometer design is limited to a frequency response below 1 kHz and therefore has been demonstrated only up to peak magnetic fields of 36 T in longer pulse magnets (~1 s duration).

The observed root-mean-square noise level of $7 \times 10^{-11}$ A m² was measured with only a 30-µs time constant and corresponds to a noise figure of $\sim 10^{-11}$ A m² Hz⁻¹/2. This measurement was not limited by the noise generated during the pulsed magnetic field, even though no special effort was taken to isolate the magnetometer from vibrations induced by the pulsed magnet. Thus, there is substantial room for improvement. We recently achieved a tenfold improvement in the resolution of capacitance measurements during the 60-T pulsed magnetic field, which reduces the measured noise figure to $\sim 10^{-13}$ A m² Hz⁻¹/2. We have also tested newer magnetometer designs, with springs 25 times as stiff ($f_{\text{res}}$ up to 50 kHz), with the goal of accessing still higher frequency quantum oscillations.

REFERENCES AND NOTES
1. There are two relevant limits: (i) the “hard spring limit,” in which our device operates, where $F = k z$ (k is the total spring constant), and (ii) the “soft spring limit,” in which $F = -\frac{md^2z}{dt^2}$ (m is the total mass of the mobile plate plus sample).
2. For example, the magnetometer can survive being dropped on the floor, although we do this as infrequently as possible.
3. Given the 500 µm by 300 µm by 1.5 µm size of that mobile plate, its mass can be calculated to be 0.31 µg, from which it was determined to be 1.2 N m⁻¹.
4. No significant change in $f_{\text{res}}$ for k was observed between room temperature and 4.2 K.
7. N. Harrison et al., ibid. 8, 5415 (1996).
12. The work of F.F.B. is supported by the U.S. Department of Energy through Los Alamos National Laboratory.

Reductive Dechlorination of DDE to DDMU in Marine Sediment Microcosms
John F. Quensen III,* Sherry A. Mueller, Mahendra K. Jain, James M. Tiedje

DDT is reductively dechlorinated to DDD and dehydrochlorinated to DDE; it has been thought that DDE is not degraded further in the environment. Laboratory experiments with DDE-containing marine sediments showed that DDE is dechlorinated to DDMU in both methanogenic and sulfidogenic microcosms and that DDD is dehydrochlorinated to DDDM three orders of magnitude more slowly. Thus, DDD does not appear to be an important precursor of the DDMU found in these sediments. These results imply that remediation decisions and risk assessments based on the calcitration of DDE in marine and estuarine sediments should be reevaluated.
contents of four replicate vials were extracted separately and analyzed (11). Some sulfate from the sediments (∼600 to 1000 μg/ml, depending on the site) was initially present in the methanogenic treatments, but this was consumed and there was no detectable sulfate present at the conclusion of the experiment at 32 weeks (13).

Table 1. Total recoveries at 32 weeks and proportions of 14C activity recovered after 32 weeks for various analytes for microcosms prepared with sediments from all three sites and under both methanogenic and sulfidogenic conditions. No methane was detected (by gas chromatography with flame ionization detection) in any of the sulfidogenic treatments.

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Sediment 3C</th>
<th>Sediment 5C</th>
<th>Sediment 8C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Live</td>
<td>Autoclaved</td>
<td>Live</td>
</tr>
<tr>
<td>Percent 14C recovered as</td>
<td>Mean SD</td>
<td>Mean SD</td>
<td>Mean SD</td>
</tr>
<tr>
<td>DDE</td>
<td>78.4 5.7</td>
<td>90.1 3.6</td>
<td>76.7 4.4</td>
</tr>
<tr>
<td>DDMU</td>
<td>12.3 2.4</td>
<td>2.0 0.3</td>
<td>11.8 1.6</td>
</tr>
<tr>
<td>TLC polar compounds</td>
<td>0.4 0.1</td>
<td>0.4 0.0</td>
<td>0.5 0.0</td>
</tr>
<tr>
<td>Aqueous phase</td>
<td>0.1 0.0</td>
<td>0.1 0.0</td>
<td>0.1 0.0</td>
</tr>
<tr>
<td>Total</td>
<td>91.2 5.6</td>
<td>92.6 3.7</td>
<td>89.1 5.6</td>
</tr>
<tr>
<td>Sulfate (μg/ml)</td>
<td>122 362</td>
<td>2446 249</td>
<td>742 243</td>
</tr>
</tbody>
</table>

Percent 14C recovered as

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Methanogenic treatments</th>
<th>Sulfidogenic treatments</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean SD</td>
<td>Mean SD</td>
</tr>
<tr>
<td>DDE</td>
<td>41.8 1.5</td>
<td>85.9 1.8</td>
</tr>
<tr>
<td>DDMU</td>
<td>45.5 2.9</td>
<td>3.1 1.5</td>
</tr>
<tr>
<td>TLC polar compounds</td>
<td>0.4 0.1</td>
<td>0.4 0.0</td>
</tr>
<tr>
<td>Aqueous phase</td>
<td>0.1 0.1</td>
<td>0.1 0.0</td>
</tr>
<tr>
<td>Total</td>
<td>87.8 1.5</td>
<td>89.4 1.4</td>
</tr>
<tr>
<td>Methane (%)</td>
<td>9.8 3.2</td>
<td>0.0 0.0</td>
</tr>
</tbody>
</table>

Transformation of DDE in all nonautoclaved microcosms was evident by thin-layer chromatography (TLC) and autoradiography (AR) of solvent extracts of the microcosms after 32 weeks of incubation (Fig. 1). The major metabolite had a relative mobility (Rf) of 0.42 and cochromatographed with an authentic standard of DDMU. In contrast, there was much less extensive transformation of DDD, mainly to more polar products. Only a trace amount of DDMU was produced from DDD. It is evident from the corresponding changes in amounts of DDE and DDMU that the DDMU was produced from DDE (Fig. 2 and Table 1).

The identity of DDMU was confirmed by gas chromatography–mass spectrometry. An authentic DDMU standard and putative DDMU in selected sample extracts gave identical retention times and mass spectra, with relative maximal abundances of 282 (DDMU parent ion) and 247, 212, and 176 m/z (mass-to-charge ratio) for fragments representing loss of one, two, and three chlorines, respectively (12).

DDMU was detected earlier and was formed more rapidly in methanogenic microcosms than in sulfidogenic ones (Fig. 3). In unamended methanogenic 3C sediment microcosms, DDMU was first quantifiable after 16 weeks of incubation, and its rate of formation between weeks 16 and 32 was 0.85 nmol per gram of sediment (dry weight) per day. In sulfidogenic treatments with 3C sediments, DDMU was not detectable until 24 weeks, and its rate of formation between weeks 16 and 32 was 0.17 nmol per gram of sediment per day. The sulfate was gradually depleted during the

![Fig. 1. Autoradiograph of TLC plate showing DDMU formed from DDE in all three sediments (3C, 5C, and 8C) but not in the sterile controls. Polar metabolites (near origin) and a trace of DDMU were formed from DDD.](image)

![Fig. 2. Mass balance between DDE dechlorination and DDMU production in the live methanogenic cultures prepared from 3C sediments. Comparable mass balance was obtained in all other treatments; there was no significant DDMU produced in any of the autoclaved control cultures (see Table 1).](image)

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incubation, but 1223 μg/ml (about half of the initial amount) still remained after 32 weeks (Table 1), and there was never any methane detected in the sulfidogenic treatments. Thus, dechlorination of DDE to DDMU was slower for the sulfidogenic treatments but did occur in the presence of sulfate.

The rate of DDMU formation (and therefore its amount at 32 weeks) varied depending on the site from which the sediment was collected (Table 1). It was greatest in 3C sediment microcosms and least in 8C sediment microcosms. In the case of 8C microcosms, the rates of DDMU formation were 0.42 and 0.10 nmol per gram of sediment per day for methanogenic and sulfidogenic conditions, respectively. These same rates were 0.36 and 0.22 nmol per gram of sediment per day for 8C sediments.

The sediments from the three sites contained 36 to 58% sand, 26 to 42% silt, 16 to 22% clay, and 3.2 to 3.6% total organic carbon. The pH of the pore water was 7.1 to 7.4; total bacterial cells, counted by epifluorescence microscopy, were 6.3 × 10⁹ to 1.3 × 10¹⁰ per gram. Concentrations of Se, Zn, Cd, Cr, Cu, Pb, Ni, and Fe were too low to adversely affect microbial activity. There were minimal differences in all of these parameters among sediments, and nothing to suggest a possible reason for the observed differences in rates and extents of DDE dechlorination among the sediments.

The results of our experiments are in contrast to the generally reported DDT degradation pathway (Fig. 4). We have found that in anaerobic marine sediments, DDE is readily dechlorinated to DDMU, and that the transformation of DDD to DDMU is relatively unimportant, occurring about three orders of magnitude more slowly.

Our results provide direct evidence for the microbial dechlorination of DDE to DDMU under anaerobic conditions and demonstrate that the results of previous studies, largely with pure cultures or with poorly controlled aeration status, cannot be used to explain the fate of DDE in anaerobic marine sediments. Thus, the notion that DDE cannot be microbially transformed should be abandoned.

Fig. 3. Time course of dechlorination of DDE to DDMU in microcosms prepared from site 3C sediment. These results were obtained by liquid scintillation counting of scappings from the TLC plates as shown in Fig. 1. The minimal background concentrations of DDE (3.4 μg/g) and DDMU (1.8 μg/g) in the 3C sediments compared with the 100 μg of 14C-DDE added per gram of sediment allowed samples from these treatments to be analyzed by gas chromatography with electron capture detection (12), and comparable results were obtained. The error bars represent sample standard deviations and where not visible are smaller than the symbols.

Fig. 4. Initial steps in the commonly accepted DDT degradation pathway (thin arrows) and steps demonstrated for Palos Verdes sediment microcosms (thick arrows). The dashed arrow indicates a minor route. Further degradation of DDMU under anaerobic conditions has been shown previously (4).

REFERENCES AND NOTES

4. DDE is generally more refractory than DDD and is shown as a terminal product in most biodegradation schemes in the literature (M. L. Rochkind and J. W. Blackburn, in Microbial Decomposition of Chlorinated Compounds [EPA-R802/88-090, U.S. Environmental Protection Agency, Washington, DC, 1986], pp. 138–145). Degradation of DDD tends to be slow, especially in aerobic soils, but its anaerobic degradation through DDMU [1-chloro-2,2-bis(p-chlorophenyl)ethylen] to DBP (p,dichlorobenzophenone) has been established for certain microbial cultures (G. Wedemeyer, Appl. Microbiol. 15, 569 (1967)).
6. The dechlorination of DDE to DDMU is analogous to the well-known microbial dechlorination of chlorinated ethanes (T. M. Vogel, C. S. Criddle, P. L. McCarty, Environ. Sci. Technol. 21, 722 (1987)). Also, the dechlorination of DDE to DDMU by zero-valent iron at environmentally relevant temperatures has been reported (G. D. Sayles, G. You, M. Wang, M. J. Kupferle, ibid. 31, 3448 (1997)).
9. Site locations. 3C: 33°43′43″N, 118°24′13″W; 5C: 33°22′11″N, 118°18′38″W; 8C: 33°41′46″N, 118°20′03″W. Site nomenclature and location were as in the Los Angeles County Sanitation District Survey of 1989 (G. J. Quensen III, J. M. Tiedje, S. A. Boyd, Appl. Environ. Microbiol. 56, 2368 (1990)).
10. Seawater base with sulfate contained the following salts (mg/liter): NaF, 3.1; SrCl₂·6H₂O, 21.0; H₂BO₃·32.4; KBr, 59.9; KCl, 745.4; CaCl₂·2H₂O, 1187; NaSO₄·13H₂O, 4307; MgCl₂·6H₂O, 5391; NaCl, 25338; Na₂SO₄·10H₂O, 21.0; Na₂HPO₄·21H₂O, 251.3. This recipe is based on that given in 40 Code of Federal Regulations, Chap. 1, Part 300, Appendix C, Section 3.2.3 (U.S. Environmental Protection Agency, Washington, DC, 1995) for the toxicity testing of estuarine species, but has been adjusted to give a total salinity of 34 parts per thousand. Seawater base without sulfate was prepared as above, except that Na₂SO₄ was omitted and an additional 3544 mg of NaCl was added per liter to maintain ionic strength. Sulfidogenic medium was prepared by mixing 1 ml of trace minerals solution and 999 ml of seawater base without sulfate. The trace minerals solution [D. R. Shelton and J. M. Tiedje, Appl. Environ. Microbiol. 47, 850 (1984)] contained the following (mg/liter): MnCl₂·4H₂O, 500; ZnCl₂·5H₂O, 40; NaMoO₄·2H₂O, 10; CoCl₂·6H₂O, 50; Na₂SeO₃·5H₂O, 50; NiCl₂·6H₂O, 50.
11. The entire contents of each vial were extracted three times by shaking for 10 min with 7 ml of petroleum ether and acetone (5:2, v:v). Solvent phases were combined and evaporated to 550 µl under a stream of dry nitrogen. Sample extracts (20 µl) were spotted on activated silica gel plates that were developed to 13 cm with 5% petroleum ether–95% hexane in a lined TLC chamber at room temperature. Autoradiography was used to determine the locations of the parent compound and metabolites on the TLC plates. Kodak Scientific Imaging Film (X-OMAT AR) was exposed to the plates (20°C to –20°C) and then developed. ART films and TLC plates were aligned on a light box, and the parent compound and metabolite zones were visualized and marked for scraping. The ¹⁴C activity in the scappings was determined by liquid scintillation counting.
12. Chromatographic conditions: 30 m DB-5 capillary column with 0.32 mm inner diameter and 0.25 µm film thickness; inlet temperature of 220°C; column temperature program of 120°C for 1 min, 30°C per minute to 180°C, 10°C per minute to 290°C, and hold for 10 min. Mass spectra were obtained by electron impact ionization.
13. Sulfate concentrations in aqueous samples were determined with a Waters Quanta 4000 Capillary Electrophoresis System equipped with a 60-cm Supelco CECel-F575 CE column (outer diameter 363 µm, inner diameter 75 µm) and ultraviolet detection at 254 nm. The retention time of sulfate was 4.8 min with Waters IonSelect Mobility Anion electrophoresis and a run voltage of 12.5 kV. Samples were diluted 1:25 and filtered through a 0.45-µm filter before analysis.
14. Supported by Montrose Chemical Corporation of California, Rhone-Poulenc, and Chris-Craft Industries. Mass spectral data were obtained at the Michigan State University Mass Spectrometry Facility, which is supported in part by a grant (DRR-00480) from the Biotechnology Program, National Center for Research Resources, National Institutes of Health.

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