of the magnetic field because they occur too quickly. This particular magnetometer has its resonant frequency at $f_{res} \sim 10$ 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 with a frequency of 615 ± 20 T, in good agreement with the accepted value of 600 ± 5 T (5–7) (Fig. 3C).

The data we report were taken on a sample roughly two orders of magnitude smaller in mass than is typical for magnetization measurements (*7*, *10*, *11*). Pulsed-field magnetization studies of two-dimensional organic compounds have long been accomplished with magnetometers composed of compensated counterwound coils (*10*). The "trampoline" magnetometer and the counterwound coil magnetometer complement each other because the latter 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 7×10^{-11} A m^2 was measured with only a 30-μs time constant and corresponds to a noise figure of $\sim 10^{-12}$ A m^2 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 m^2 Hz$^{-1/2}$. We have also tested newer magnetometer designs, with springs 25 times as stiff (f_{res} up to 50 kHz), with the goal of accessing still higher frequency quantum oscillations.

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1. There are two relevant limits: (i) the "hard spring limit," in which our device operates, where $\mathbf{F} = k\,\delta\mathbf{z}$ (k is the total spring constant), and (ii) the "soft spring limit," in which $\mathbf{F} = md^2\mathbf{z}/dt^2$ (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 300 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 k was determined to be 1.2 N m^{-1}. No significant change in f_{res} or k was observed between room temperature and 4.2 K.
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Reductive Dechlorination of DDE to DDMU in Marine Sediment Microcosms

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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 DDMU 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 recalcitrance of DDE in marine and estuarine sediments should be reevaluated.

DDT [1,1,1-trichloro-2,2,bis(*p*-chlorophenyl)ethane] was one of the first synthetic pesticides to gain wide acceptance. Initially its use greatly enhanced crop yields, but pest species rapidly developed resistance so that its use in agriculture in the United States began to decline by 1959. It was effective longer in controlling mosquito-borne malaria (*1*) and is still used for that purpose in some tropical countries.

Because of environmental concerns, the use of DDT was banned in the United States and in some other countries in the early 1970s. By that time, however, it was distributed globally. Both DDD [1,1-dichloro-2,2,-bis(*p*-chlorophenyl)ethane] and DDE [1,1-dichloro-2,2,-bis(*p*-chlorophenyl)-ethylene] existed as by-products in commercial DDT formulations, and both may be formed by environmental degradation of DDT. DDT, DDD, and DDE (collectively DDX) are found, in various proportions, in soils and sediments and have been reported at 3422 out of 22,000 sites identified as posing a danger to humans and animal life by the U.S. Environmental Protection Agency (EPA) in its National Sediment Quality Survey.

One such marine site is the continental shelf off of the Palos Verdes Peninsula in southern California. Sediment core data collected over the last two decades by the Los Angeles County Sanitation District and the U.S. Geological Survey show that DDE is the most prevalent of the DDX compounds present in the shelf sediments and imply that the mass of DDE is decreasing with time (*2*, *3*). Bioturbation has been proposed as a mechanism responsible for this trend (*3*) because DDE is viewed as a recalcitrant compound (*4*). However, the concentration of trace metals in the sediments has remained constant with time, which is not consistent with bioturbation (*5*), and there is precedence for believing that DDE can be reductively dechlorinated to DDMU (*6*), which is also found in the sediment (*2*, *3*). Thus, an alternative explanation for the disappearance of DDE from the sediments, first proposed by List (*7*), is that it is being reductively dechlorinated to DDMU by

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Table 1. Total recoveries at 32 weeks and proportions of ^{14}C 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. 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*).

Analyte	Sediment 3C				Sediment 5C				Sediment 8C			
	Live		Autoclaved		Live		Autoclaved		Live		Autoclaved	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Sulfidogenic treatments												
Percent ^{14}C recovered as												
DDE	78.4	5.7	90.1	3.6	76.7	4.4	86.7	6.0	80.5	7.7	86.9	2.7
DDMU	12.3	2.4	2.0	0.3	11.8	1.6	3.0	1.8	5.5	2.1	3.0	2.6
TLC polar compounds	0.4	0.1	0.4	0.0	0.5	0.0	0.4	0.1	0.4	0.0	0.4	0.0
Aqueous phase	0.1	0.0	0.1	0.0	0.1	0.0	0.1	0.0	0.1	0.0	0.1	0.0
Total	91.2	5.6	92.6	3.7	89.1	5.6	90.2	5.3	86.2	5.8	90.5	2.2
Sulfate (μg/ml)	1223	326	2446	249	742	243	2654	19	1123	119	2738	97
Methanogenic treatments												
Percent ^{14}C recovered as												
DDE	41.8	1.5	85.9	1.8	61.1	5.8	86.6	1.0	71.9	1.8	89.8	3.5
DDMU	45.5	2.9	3.1	1.5	24.0	2.8	2.5	1.9	18.1	2.9	2.9	2.7
TLC polar compounds	0.4	0.1	0.4	0.0	0.3	0.0	0.5	0.3	0.3	0.0	0.5	0.2
Aqueous phase	0.1	0.1	0.1	0.0	0.1	0.0	0.1	0.0	0.1	0.0	0.1	0.0
Total	87.8	1.5	89.4	1.4	85.5	5.5	89.8	2.5	90.4	3.5	93.2	1.3
Methane (%)	9.8	3.2	0.0	0.0	8.4	5.6	0.0	0.0	6.0	4.4	0.0	0.0

anaerobic marine bacteria in the same manner as PCBs (polychlorinated biphenyls) (*8*). We therefore reexamined the possible fates of DDE and DDD in these marine sediments to determine whether DDE or DDD is the more probable precursor of the DDMU currently found in the sediment.

We prepared sediment slurries by mixing equal volumes of Palos Verdes shelf sediments from one of three locations (3C, 5C, or 8C) (*9*) with one of two anaerobic marine media. For sulfidogenic treatments, the medium contained the same amount of sulfate (27 mM) normally found in seawater (*10*). For methanogenic treatments, the sulfate was omitted, and the ionic strength of the medium was maintained by increasing the amount of sodium chloride. The individual microcosms consisted of 7 ml of sediment slurry (containing ~2 g of sediment on a dry weight basis), spiked with 200 μg of ^{14}C-labeled DDD or DDE in 7 μl of acetone, in a tightly stoppered 20-ml glass vial. Autoclaved slurries served as controls for nonbiological effects. The microcosms were mixed thoroughly after addition of the DDD or DDE and then incubated statically at room temperature (22° to 25°C). At 8-week intervals, the entire contents of four replicate vials were extracted separately and analyzed (*11*).

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 (R_f) 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

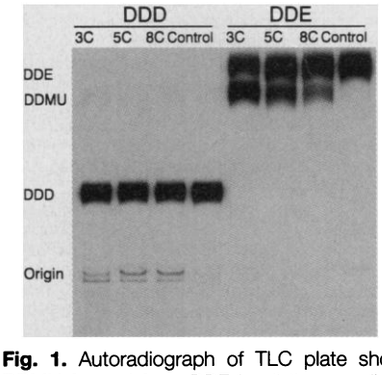

Fig. 1. Autoradiograph of TLC plate showing DDMU formed from DDE in all three sediments (3C, 5C, and 8C) but not in the sterile controls. Polar metabolites (near origin) and a trace of DDMU were formed from DDD.

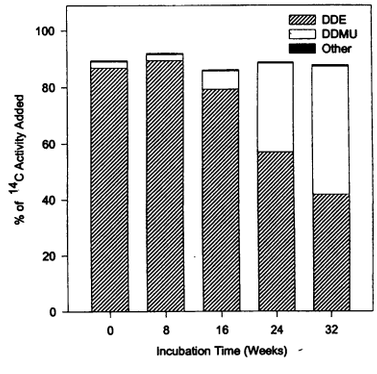

Fig. 2. Mass balance between DDE dechlorination and DDMU production in the live methanogenic cultures prepared from 3C sediments. Comparable mass balance was obtained in all other treatments; there was no significant DDMU produced in any of the autoclaved control cultures (see Table 1).

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 5C sediments.

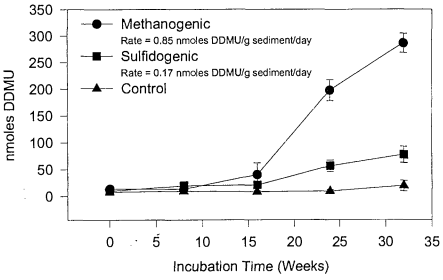

Fig. 3. Time course of dechlorination of DDE to DDMU in microcosms prepared from site 3C sediment. These results were obtained by liquid scintillation counting of scrapings from the TLC plates as shown in Fig. 1. The minimal background concentrations of DDE (3.4 μg/g) and DDMU (1.8 μg/g) in the 3C sediments 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.

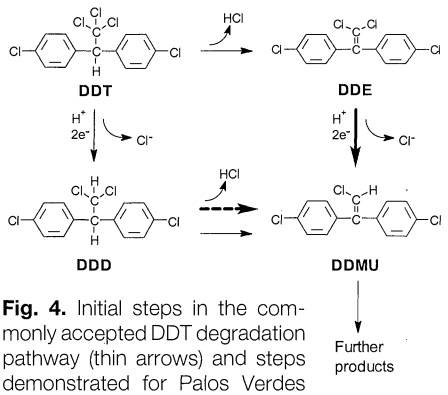

Fig. 4. Initial steps in the commonly accepted DDT degradation pathway (thin arrows) and steps demonstrated for Palos Verdes sediment microcosms (thick arrows). The dashed arrow indicates a minor route. Further degradation of DDMU under anaerobic conditions has been shown previously (*4*).

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 6.3×10^9 to 1.3×10^{10} 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.

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9. Site locations. 3C: 33°43.843′N, 118°24.133′W; 5C: 33°42.913′N, 118°21.893′W; 8C: 33°41.846′N, 118°20.003′W. Site nomenclature and location were as in the Los Angeles County Sanitation District Survey of 1989 (*2*, *3*). Sediments were brought to the surface with a box core and subsampled with a polyvinyl chloride pipe 10 cm in diameter. The pipe ends were tightly stoppered; the cores were then transported on ice to the laboratory and stored at 4°C until use (within 3 months of collection). Sediments, slurries, and sample vials were purged with filter-sterilized, O_2-free N_2-CO_2 (80:20, v:v) during experiment set-up by a Hungate gassing apparatus [R. E. Hungate, *Adv. Microbiol.* **3B**, 117 (1968)].
10. Seawater base with sulfate contained the following salts (mg/liter): NaF, 3.1; $SrCl_2 \cdot 6H_2O$, 21.0; H_3BO_3, 32.4; KBr, 59.9; KCl, 754.5; $CaCl_2 \cdot 2H_2O$, 1187; Na_2SO_4, 4307; $MgCl_2 \cdot 6H_2O$, 5391; NaCl, 25338; $Na_2SiO_3 \cdot 9H_2O$, 21.0; $NaHCO_3$, 215.3. 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 Na_2SO_4 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 with sulfate. Methanogenic 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): $MnCl_2 \cdot 4H_2O$, 500; $ZnCl_2$, 50; $CuCl_2 \cdot 2H_2O$, 40; $NaMoO_4 \cdot 2H_2O$, 10; $CoCl_2 \cdot 6H_2O$, 50; Na_2SeO_3, 50; $NiCl_2 \cdot 6H_2O$, 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 500 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 TLC plates for 6 days at −20°C and then developed. AR films and TLC plates were aligned on a light box, and the parent compound and metabolite zones were visualized and marked for scraping. The ^{14}C activity in the scrapings 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 CElect-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 electrolyte 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 Research Technology Program, National Center for Research Resources, National Institutes of Health.

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