Table I. Catalytic Properties of Antibodies

IgG	k_{cat}(**2a**) (min^{-1} × 10^3)	K_m(**2a**) (μM)	K_i(**1**) (μM)	k_{cat}/ [k_{OH^-}[OH$^-$]]	k_{2b}/k_{2a}
6E4D5	2.3 ± 0.3	4.4 ± 0.5	0.5 ± 0.1	33	<0.005
3E10D8	7.0 ± 0.7	6.0 ± 0.6	0.8 ± 0.2	100	0.025
3E9F2D10	5.8 ± 0.6	4.5 ± 0.5	1.6 ± 0.4	83	0.018
2H12E4	18.7 ± 2.0	14.8 ± 1.0	2.4 ± 0.6	267	<0.005
2B5B11	9.8 ± 1.0	6.2 ± 0.6	1.4 ± 0.3	140	<0.005

3 to a measurable extent under a variety of conditions, 18 antibodies were found to catalyze the hydrolysis of the corresponding ester **2**.

Interestingly, all of the 18 antibodies found to accelerate the hydrolysis of ester **2** were selective for the D-phenylalanine-containing diastereomer, **2a**, although a mixture of diastereomers was used to elicit the antibodies. One explanation is that haptens containing D amino acids (or analogues thereof) are more immunogenic than those containing L amino acids.[15] Of these 18 catalytic antibodies, five IgGs were selected for further study. All five exhibited saturation kinetics and competitive inhibition by phosphonate **1**, as determined by Eadie–Hofstee and Dixon plots, respectively (Table I).[16] With all the antibodies, multiple (>10) turnovers were observed with no loss of catalytic activity. Because the antibody-catalyzed reaction rate is first-order in hydroxide ion concentration (vide infra), the value of k_{cat} can be compared with k_{OH^-}[OH$^-$] for the hydrolysis of ester **2a** to give the rate enhancements (k_{cat}/k_{OH^-}[OH$^-$]) shown in Table I.[17] In other antibody-catalyzed reactions, greater rate enhancements can be attributed to a higher degree of differential binding of the antibody to transition state vs ground state and/or the presence of catalytic groups in the antibody combining site. With relatively large transition state analogues such as these tripeptides, the tetrahedral phosphonate probably contributes proportionally less to the overall binding energy of the hapten to the antibody.

The antibody-catalyzed reaction shows a high degree of stereospecificity for the D-phenylalanine-containing ester **2a**. With all five of the antibodies studied, hydrolysis of the L-phenylalanine-containing ester **2b** (30 μM) was catalyzed at <2.5% the rate of **2a** (initial rates) (Table I). With three of the five IgGs, the ratio of **2b**:**2a** hydrolysis rates was less than 0.5%. This value represents the upper limit on the ratio of the **2b**:**2a** rates, since the **2b** rate with these antibodies was essentially indistinguishable from the background hydrolysis rate at pH 8.0. The antibodies show a high degree of substrate specificity. Esters **4** and **5**, containing leucine or tryptophan, respectively, in place of phenylalanine, are not hydrolyzed by any of the antibodies (k_4/k_{2a} < 0.005 and k_5/k_{2a} < 0.005 at 30 μM substrates, pH 8.0, 24 °C).

The catalytic activity of antibodies 6E4D5, 2H12E4, and 2B5B11 shows a linear dependence on hydroxide ion concentration in the range of pH 7–10.[18] Interestingly, with antibodies 3E10D8 and 3E9F2D10, the pH dependence shows an inflection at pH 9.2. This result suggests the presence of a catalytic amino acid side chain in these two antibodies, such as a tyrosine residue, which could act as a nucleophile, producing a labile tyrosine ester intermediate. Chemical modification of tyrosine residues with tetranitromethane[19] destroyed the catalytic activity of all the antibodies.

We have described the generation of antibodies which catalyze the stereospecific hydrolysis of esters, a potentially useful class of organic reactions for synthetic chemistry. In addition to providing new biological catalysts, these rationally designed antibodies may suggest clues to the evolution and mechanism of enzymes.

Acknowledgment. We acknowledge the Affymax Research Institute and the Office of Naval Research (N00014-87-K-0256) for support.

Supplementary Material Available: Synthesis details and characterization (mp, IR, ^{1}H NMR, MS) of compounds **1**–**5** (17 pages). Ordering information is given on any current masthead page.

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(14) IgGs were purified from ascites fluid by affinity chromatography using protein A coupled Sepharose 4B[22] followed by Pharmacia Mono Q 10/10 anion exchange chromatography using a linear gradient of 85–150 mM sodium chloride (20 mM Tris, pH 7.8) over 20 min (flow rate of 4.0 mL/min). The antibody-containing fractions were dialyzed exhaustively against assay buffer and judged to be homogeneous by 12.5% sodium dodecyl sulfate polyacrylamide gel electrophoresis with Coomasie blue staining.[23]

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(17) The rate $v_{uncat} = k_{OH^-}$[**2a**][OH$^-$] was compared directly to the rate of hydrolysis in the IgG–substrate complex, $v_{complex} = k_{complex}$[complex][OH$^-$]. The value of the pseudo-first-order rate constant k_{OH^-}[OH$^-$] was determined to be (7.0 ± 0.2) × 10^{-5} min^{-1} at 24 °C, pH 8.0, by extrapolation of the rate of the uncatalyzed reaction to zero buffer concentration.

(18) The rates of hydrolysis were measured at 10 different pH values between pH 7 and 10, in BBS, at 24 °C, with 50 μM **2a** (approximately V_{max} conditions). The slope of log (v_{obs}) vs pH was 0.82 ± 0.05 for all five IgGs except where the rate levelled off at high pH with 3E10D8 and 3E9F2D10 as noted in the text.

(19) The antibodies (20 μM) were treated with 2.0 mM tetranitromethane[24] in BBS, pH 8, for 1 h at 25 °C, followed by dialysis against BBS. In separate experiments, hapten **1** (250 μM) was also present. Greater than 90% of the catalytic activity was retained in the latter cases.

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Infinite Polymeric Frameworks Consisting of Three Dimensionally Linked Rod-like Segments

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We propose that a new and potentially extensive class of solid polymeric materials with unprecedented and possibly useful properties may be afforded by linking together centers with either a tetrahedral or an octahedral array of valencies by rod-like connecting units. For example, if ways could be devised of linking together tetrahedral centers and appropriate molecular rods, an infinite molecule would be generated which might be expected to adopt a structure based on either the cubic diamond lattice or the hexagonal Lonsdaleite lattice in which each C–C bond of the prototype had been replaced by a molecular rod. These frameworks would generate a regular array of cavities, interconnected by windows. When models of 3D networks based on tetrahedral centers are constructed, a very striking feature is the relatively large size of the cavities and windows produced even by rods of only modest length.

Lattices of this general type seemed to us to offer a number of features of sufficient potential interest and utility to warrant attempting the synthesis of some exploratory examples. For instance, they may show interesting molecular sieve and ion exchange properties, they may have unusual mechanical and elec-

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trical properties, and they may, after appropriate functionalization of the rods, provide tailor-made materials for the heterogeneous catalysis of a wide range of transformations.

We describe here the synthesis and X-ray crystal structure of the first example of a deliberately designed and constructed infinite framework consisting of tetrahedral centers linked together by rod-like units. An enormous range of different approaches to such frameworks can be conceived; the particular approach described here involves substitution of the acetonitrile ligands in $Cu^I(CH_3CN)_4^+$ by 4,4′,4″,4‴-tetracyanotetraphenylmethane, I.

The classical bromination catalyzed by iron (2.55 g) of tetraphenylmethane (12.3 g) with Br_2 (29 g) in CCl_4 (100 mL) was used to obtain 4,4′,4″,4‴-tetrabromotetraphenylmethane, which was recrystallized from xylene or DMF [^1H NMR ($CDCl_3$), very clean symmetrical 12 component AA′BB′ pattern indicative of para substitution, with the major "quartet" components at 7.44, 7.35, 7.05, and 6.96 ppm. Anal. Calcd for $C_{25}H_{16}Br_4$: Br, 50.3. Found: Br, 50.3]. The tetrabromo derivative was converted to I by reaction with CuCN (0.97 g) in refluxing DMF (2.6 mL) for 4 h.[1] The product was purified by chromatography on silica gel (Merck Kieselgel 60) with chloroform as eluent. Recrystallization from dichloromethane–benzene, followed by drying at 100 °C in vacuum, gave I [Anal. Calcd for $C_{29}H_{16}N_4$: C, 82.8; H, 3.8; N, 13.3. Found: C, 82.7; H, 3.7; N, 13.0. ^1H NMR ($CDCl_3$), AA′BB′ pattern with major "quartet" components at 7.69, 7.60, 7.32, and 7.23 ppm. MS, *m/e* (70 eV, EI), 420 (M^+, 66.4%), 318 ($M^+ - C_6H_4CN$, 100%)]. Slow evaporation of a solution in nitrobenzene (10 mL) and acetonitrile (1 mL) of $Cu(CH_3CN)_4BF_4$[2] (22×10^{-3} g) and I·nC_6H_6 (n = ca. $^1/_2$) (40×10^{-3} g) gave colorless crystals of $Cu^I[C(C_6H_4{\cdot}CN)_4]BF_4{\cdot}xC_6H_5NO_2$ some of which were set aside for crystallographic analysis and kept moist with mother liquor. The remaining crystals were collected, washed with nitrobenzene, and dried in a stream of air until a free-running product was obtained. The product in this form lost 65.4% by weight upon drying at 100 °C under vacuum [Anal. of dried residue. Calcd for $C_{29}H_{16}BCuF_4N_4$: C, 61.0; H, 2.8; N, 9.8; Cu, 11.1. Found: C, 60.9; H, 3.1; N, 9.5; Cu, 11.3]. The weight loss of 65.4% corresponds to a value of x in the above formulation of ca. 8.8. The IR spectrum (KBr disc) of the above air-dried, free-running solid showed a very sharp $\nu_{C\equiv N}$ at 2240 cm^{-1}, dominant nitrobenzene bands at 705, 1345, and 1520 cm^{-1}, and a strong BF_4^- band at 1060 cm^{-1}, positions of other bands arising from the tetranitrile moiety and from the nitrobenzene were very similar to those observed for I (KBr disc) and nitrobenzene (liquid film) themselves.

A crystal was sealed with mother liquor in a Lindemann tube for X-ray crystallographic analysis.[3] Positions of Cu atoms and all the C and N atoms of the $C(C_6H_4{\cdot}CN)_4$ units were immediately revealed in the Patterson map for the space group $I\bar{4}m2$ and in subsequent electron density maps these atoms were very well resolved. By contrast, no conclusive evidence was found for positions of any of the atoms associated with either the BF_4^- anions or the nitrobenzene molecules, indicating a largely random distribution of these species. The structure consists of an infinite, positively charged, diamond-related C/N/Cu framework as shown in Figure 1 with disordered, essentially "liquid" nitrobenzene together with BF_4^- ions in the insterstitial space. We estimate that the framework occupies only approximately one-third of the volume of the crystal, the remaining two-thirds being occupied by at least $7.7C_6H_5NO_2$ per Cu together with mobile BF_4^- ions. Regions of attenuated electron density of ca. 0.6 e $Å^{-3}$ were detected in the intraframework spaces which suggests some slight ordering of the largely disordered nonframework species, which we are unable to model. This small degree of ordering of a major fraction of the contents of the crystal accounts for the high R value of 0.17, which gives a misleadingly poor impression of the reliability of the structure of the ordered, minor fraction of the crystal, namely the framework. Within the framework, the interatomic distances, the bond angles, and the entire three-dimensional disposition arising from the analysis are completely in accord with the intended structure and with chemical common sense. Despite the high R value the structure of the framework is beyond doubt. We find it astonishing that a material, such a large fraction of which is essentially liquid, provides a diffraction pattern at all; under the circumstances the high R value is not surprising.

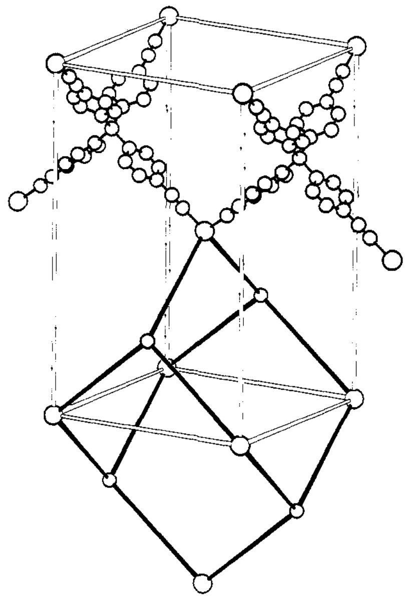

Figure 1. Structure of $\{Cu^I[C(C_6H_4{\cdot}CN)_4]\}_n^{n+}$ framework. One complete tetragonal unit cell together with parts of adjoining cells are shown; the larger circles denote the copper atoms. All $C{\cdot}C_6H_4{\cdot}CN{\cdot}Cu$ rods are 8.856 (2) Å long. An adamantane-like cavity with its center at the center of the bottom face of the complete unit cell is highlighted. Bond lengths: CuN, 2.03 (4) Å; C≡N, 1.05 (6) Å; CC distances normal.

The framework generates large tetragonally elongated adamantane-like cavities, one of which is highlighted in Figure 1. The volume of each adamantane cavity defined by the van der Waals surfaces of the surrounding rods is approximately 700 $Å^3$. Two different adamantane-like cavities are present, one type bounded by six tetrahedral copper centers and four tetrahedral carbon centers, the other by six carbons and four coppers. The composition of the material requires that there are twice as many adamantane cavities as BF_4^- ions. Presumably anions would prefer to occupy the more positively charged Cu_6C_4 cavities, but no crystallographic evidence for the location of the BF_4^- ions was forthcoming because of their apparent freedom of migration within the framework. All copper and tetrahedral carbon centers are located at sites of symmetry $\bar{4}2m$. Each of the mirror planes intersecting at these sites also passes through two of the four associated C_6H_4CN residues such that the C_6H_4 rings are perpendicular to the mirror plane.

After 1 h at room temperature suspended in a solution of NBu_4PF_6 (large excess) in nitrobenzene the crystals appeared, under the microscope, to retain their external faces and edges and their internal transparency; however, the IR spectrum of the product showed complete replacement of the intially strong BF_4^- band at 1060 cm^{-1} by an intense band at 830 cm^{-1} together with a medium band at 560 cm^{-1}, both associated with PF_6^-, the rest of the spectrum being essentially unchanged. It remains to be seen whether or not the ordered framework is retained intact after single crystals are subjected either to removal and exchange of interstitial solvent or to anion exchange.

In summary, this work establishes the general feasibility of constructing deliberately designed infinite molecules consisting of three dimensionally linked rod-like segments and reveals an unusual situation in which approximately two-thirds of the contents of what is undoubtedly a crystal are effectively liquid.

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Supplementary Material Available: Crystal data and data collection, structure determination and refinement, numbering scheme, and tables of crystal data and structure determination, fractional atomic coordinates and isotropic thermal parameters, and interatomic distances and angles for $Cu[C(C_6H_4{\cdot}CN)_4]BF_4{\cdot}xC_6H_5NO_2$ (6 pages); listing of observed and calculated structure factors (3 pages). Ordering information is given on any current masthead page.

(−)-α,α-Dichlorocamphorsulfonyloxaziridine: A Superior Reagent for the Asymmetric Oxidation of Sulfides to Sulfoxides[†]

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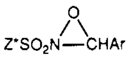
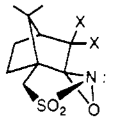
Optically active sulfoxides are important synthons for the asymmetric construction of C–C bonds[1,2] and have played pivotal roles in studies of the origins of asymmetric induction.[2,3] The Andersen procedure, the reaction of an organometallic reagent with a chiral sulfinate ester, is the method of choice for synthesis of most enantiomerically pure sulfoxides. However, an attractive alternative is the asymmetric oxidation of prochiral sulfides to sulfoxides with optically active oxidizing reagents.[2,4] Not only can the sulfoxide be formed in one step but also the synthesis of optically active sulfoxides, not possible by the Andersen procedure, can be realized. Unfortunately, the stereoselectivities for the asymmetric oxidizing reagents developed to date are variable, being both reagent and substrate dependent.[2-4]

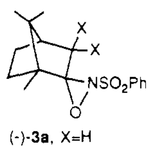
The modified Sharpless reagent,[5,6] developed by Kagan et al., is the most effective of these reagents, affording moderate to excellent stereoselectivities for a variety of sulfides substrates (Table I).[6] The enantiomerically pure *N*-sulfonyloxaziridines **1** and **2a** are another class of asymmetric oxidizing reagents which are aprotic in nature.[4,7,8] The highest stereoselectivities (31–91% ee) are observed for the *N*-sulfamyloxaziridines **1b**,[4] which is unfortunate because (+)-(camphorylsulfonyl)oxaziridine (**2a**) is easily prepared and separation of the oxaziridine diastereoisomers, a problem with oxaziridines of type **1**, is not necessary for **2**.[7] While the α,α-dichloro and oxo derivatives, **2b** (56–74% ee)[9] and **2c** (5–62% ee),[10] afford improved stereoselectivities over **2a** (3–80% ee),[7] for sulfide oxidations these reagents are also substrate specific.



(S,S)-**1a**, **Z***= (+)-camphor
b, **Z***= (-)(S)-N-(α-methylbenzyl)-N-benzylamine



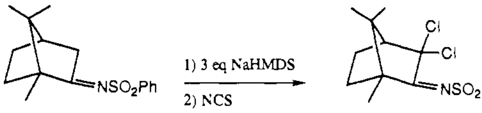
(+)-**2a**, X=H
(-)-**2b**, X=Cl
(+)-**2c**, X=O



(-)-**3a**, X=H
(-)-**3b**, X=Cl

In this context we report that (−)-α,α-dichlorocamphorsulfonyloxaziridine (**3b**), a new class of *N*-sulfonyloxaziridines, is a highly efficient reagent for the asymmetric oxidation of sulfides to sulfoxides (68–>95% ee) and is also remarkably general (Table I).

(−)-Camphorsulfonimine **4** is readily prepared in 70% yield by refluxing equivalent amounts (100 mmol) of (+)-camphor and benzenesulfonamide with 0.5 equiv of titanium tetrachloride and 3 equiv of triethylamine in 1,1,2-trichloroethylene for 20 h.[11-13] Treatment of (−)-**4** with 3 equiv of bis(trimethylsilyl)amide (NHMDS) at −78 °C followed by addition of the azaenolate[14] to 3 equiv of *N*-chlorosuccinimide (NCS) gave (−)-**5** in 80% isolated yield.[13] Biphasic oxidation of sulfonimines (−)-**4** and (−)-**5**, with m-CPBA/saturated K_2CO_3 as previously described,[14] gives oxaziridines **3a–b** in 87 and 75% isolated yield, respectively, as single isomers following flash chromatography.[15] While oxidation of **4** to **3a** is complete within 4 h, **5** requires up to 4 days, reflecting the greater steric hindrance of the C–N double bond toward oxidation.



(-)-**4** (-)-**5**

Asymmetric oxidations were carried out by treating equivalent amounts of the sulfide with **3a–b** (typically 0.5–1.0 mmol) as outlined in Table I. The sulfoxides were isolated by preparative TLC, and the optical purities were determined by using Eu(hfc) and as described in Table I. The sulfonimine reduction products (−)-**4** and (−)-**5** were recovered in 80–90% yield and recycled.

(−)-α,α-Dichlorocamphorsulfonyloxaziridine (**3b**) in CCl_4 affords uniformly high stereoselectivities (66–>95% ee) for the asymmetric oxidation of alkyl aryl sulfides (entries 2, 4, 6, 7, and 9), functionalized sulfides (entries 10–13), and for a dialkyl sulfide (entry 14). Significantly, a number of the sulfoxides obtained in very high optical purity (entries 2, 9, 11, 13, and 14) are useful chiral synthons for the synthesis of enantiomerically pure compounds (EPC).[1,2] Oxidation using **3b** in most cases gave higher ee's than the modified Sharpless reagent. Lower ee's were observed for oxaziridine **3a** and for **3b** in CH_2Cl_2.

In 1983 we demonstrated that the configurational lability of the selenoxide moiety is the result of acid-catalyzed achiral hydrate formation.[16,17] For this reason enantiomerically pure *N*-

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[†] The Chemical Abstracts Service name for the title compound (**3b**) is 3,3-dichloro-1,7,7-trimethyl-2′-(phenylsulfonyl)spiro[bicyclo[2.2.1]heptane-2,3′-oxaziridine]. The CAS name for **2a** is tetrahydro-9,9-dimethyl-4*H*-4a,7-methanooxazirino[3,2-*i*][2,1]benzisothiazole 3,3-dioxide.

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(11) Gosciniak, D. J. Ph.D. Dissertation, Drexel University, 1984.

(12) Independently, Jennings and Lovely described a similar method for the synthesis of camphorsulfonimines, see: Jennings, W. B.; Lovely, C. J. *Tetrahedron Lett.* **1988**, 3725.

(13) The sulfonimines isolated by flash chromatography had the following properties: (−)-**4**, mp 92–3 °C (EtOH); $[\alpha]_D^{20}$ −35.8° (*c* 3.0 $CHCl_3$); (−)-**5**, mp 141–2 °C (EtOH); $[\alpha]_D^{20}$ −28.7° (*c* 2.9 $CHCl_3$). All new compounds gave satisfactory elemental analysis.

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(15) Properties of the oxaziridines: **3a**, mp 133–5 °C (EtOH); $[\alpha]_D^{20}$ −198° (*c* 3.0 $CHCl_3$); **3b**, mp 121–2 °C (EtOH); $[\alpha]_D^{20}$ −150° (*c* 4.2 $CHCl_3$).

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