two molecules of bis(1-methylimidazolyl)ketone, methylation of the resulting diol, and deprotection.¹¹ Treatment of 1 in MeOH with 2 equiv of $Cu(OAc)_2 H_2O$ and an excess of $(Bu_4N)(ClO_4)$ caused the formation of a blue precipitate, which after crystallization from CH₃CN/THF yielded [Cu₂(1)(μ -OH)(μ -OAc)]- $(ClO_4)_2$ ·1.5THF (2·1.5THF) as blue plates (80%).¹² Crystallization from MeOH/THF afforded X-ray quality crystals of 2.1.5THF.MeOH, the solid-state structure of which was determined (Figure 1).¹³

Molecule 2 has crystallographically required C_s symmetry, with the mirror plane bisecting the phenyl ring and the hydroxo and acetato bridges. Only four of the six imidazole groups in 1 are coordinated to copper atoms, each of which adopts a D_{2d} distorted square-planar geometry. The dihedral angle between the best least-squares planes through the two copper coordination spheres is 62.5°. Perchlorate counterions, hydrogen bonded to the uncoordinated imidazoles at N(11), and THF molecules are accommodated in the crystal lattice. Most interesting is a methanol solvent molecule located on the mirror plane and inserted into the cavity defined by the bis(imidazolyl)benzene linker. The resulting inclusion compound¹⁴ resembles the vaulted lacunar cyclidenes studied by Busch and co-workers.¹⁵ The location of O(5) in axial positions, the O(5)-Cu(Cu') distance being 2.59 (1) Å, suggests that inclusion of methanol is partly derived from weak ligation to the copper atoms. Alternative explanations, such as hydrogen bonding either to the uncoordinated imidazole or the bridging hydroxide ion, are precluded by the long O(5)-N(13) distance [3.34 (1) Å] and the location of the methanol opposite to the hydroxo bridge.

The X-band ESR spectrum of a frozen solution of 2-1.5THF in CH₃CN at 12 K has axial features ($g_{\parallel} = 2.33, g_{\perp} = 2.08, A_{\parallel}^{Cu}$ = 1.90 (5) × 10⁻² cm⁻¹) characteristic of N₂O₂ coordination geometries in natural copper proteins.¹⁶ In addition, we observe a $|\Delta M_{\rm S}| = 2$ transition at $g \sim 4.3$, suggesting the presence of spin exchange coupling.¹⁷ Solid-state magnetic susceptibility data for 2-1.5THF, obtained between 6-300 K on a SQUID magnetometer, were fit to the Bleaney-Bowers expression.¹⁸ The weak ferromagnetic coupling observed, J = +1.3 (1) cm⁻¹, differs from the moderate-to-strong antiferromagnetic interactions reported for many other hydroxo-bridged dicopper(II) complexes^{19,20} but falls

(10) (a) Synthesized via the method of Oxley and Short (Oxley, P.; Short, (10) (a) Synthesized via the method of Oxiey and Short (Oxiey, r., Short, W. F. J. Chem. Soc. 1947, 497).
 (b) Hughey, J. L.; Knapp, S.; Schugar, H. Synthesis 1980, 489.
 (c) Knapp, S.; Keenan, T. P.; Zhang, X.; Fikar, R.; Potenza, J. A.; Schugar, H. J. J. Am. Chem. Soc. 1987, 109, 1882.
 (d) Traylor, T. U.; Hill, K. W.; Tian, Z.-Q.; Rheingold, A. L.; Peisach, J.; McCracken, J. Ibid. 1988, 110, 5571.
 (e) Lipshutz, B. M.; Vaccaro, W.; Huff, D. & Tarabadom Lett. 1996.
 (f) Whitten, L. P. Matthews, D. P. B. Tetrahedron Lett. 1986, 27, 4095. (f) Whitten, J. P.; Matthews, D. P.; McCarthy, J. R. J. Org. Chem. 1986, 51, 1891.

1) Further details of the preparation of 1 will be provided elsewhere. ¹H and ¹³C¹H NMR, FTIR, and FAB-MS spectral data for 1 are included as Supplementary Material.

(12) Anal. (C₄₀H₅₀N₁₂O_{14.5}Cl₂Cu₂): C, H, N, Cl; FTIR (Nujol, cm⁻¹) (12) Anal. (C₄₀H₅₀N₁₂O₁₄₅Cl₂Cu₂): C, H, N, Cl; FTIR (Nujol, cm⁻¹) 3563 (OH) (OD band obscured by Nujol envelope), 3225 (NH) [2405 (ND)], 3063, 2900-3000, 1570, 1570, 1468, 1449, 1427, 1284, 1103 (ClO₄), 1027, 989, 908, 807, 761, 710, 691, 624 (ClO₄) cm⁻¹; UV-vis (CH₃CN) [λ_{max} , nm (ϵ_M /Cu cm⁻¹ M⁻¹]] 274 (17700), 290 (sh), 315 (sh), 617 (74); magnetic susceptibility, solution (294 K, 2.5 mM in CD₃CN) 1.7 μ_B /Cu. (13) Crystal data for **2**-1.5THF-MeOH (C₄₁H₅₄N₁₂O_{15.5}Cl₂Cu₂, M_r = 1160.94) at 296 K: size 0.46 × 0.26 × 0.11 mm, monoclinic, space group P_{2_1}/m (no. 11), a = 8.529 (4) Å, b = 27.883 (4) Å, c = 11.133 (5) Å, $\beta =$ 102.81 (2)°, V = 2582 (3) Å³, Z = 2, $\rho_{calid} = 1.493$ g, cm⁻³, $\rho_{meas} = 1.49$ (1) g cm⁻³. For 2028 unique, observed reflections with $F^2 > 3\sigma(F^2)$ and 349 variable parameters. R = 0.065 and $R_{-2} = 0.080$.

variable parameters, R = 0.065 and R_w = 0.080.
 (14) (a) Cram, D. J. Angew. Chem., Int. Ed. Engl. 1988, 27, 1009. (b)

Lehn, J.-M. Ibid. 1988, 27, 89.

(15) Ramprasad, D.; Lin, W.-K.; Goldsby, K. A.; Busch, D. H. J. Am. Chem. Soc. 1988, 110, 1480.

(16) Peisach, J.; Blumberg, W. E. Arch. Biochem. Biophys. 1974, 165, 691. (17) Davis, W. M.; Lippard, S. J. Inorg. Chem. 1985, 24, 3688 and references therein.

 (18) Bleaney, B.; Bowers, K. D. Proc. R. Soc. London, A 1952, 214, 451.
 (19) A notable exception: Fallon, G. D.; Murray, K. S.; Spethmann, B.; Yandell, J. K.; Hodgkin, J. H.; Loft, B. C. J. Chem. Soc., Chem. Commun. 1984, 1561.

(20) (a) Coughlin, P. K.; Lippard, S. J. J. Am. Chem. Soc. 1984, 106, 2328. (b) Benzekri, A.; Dubourdeaux, P.; Latour, J.-M.; Laugier, J.; Rey, P. Inorg. Chem. 1988, 27, 3710 and references therein. (c) Bertocello, K.; Fallon, G. D.; Hodgkin, J. H.; Murray, K. S. Ibid. 1988, 27, 4750 and references therein.

within the range of values found for (µ-alkoxo)(µ-acetato)dicopper(II) species.¹⁷ Consistent with the small J value, the effective moment in solution at 294 K²¹ is 1.7 μ_B per copper, close to the spin only value.

Encouraged by the dinucleating ability of 1, as manifested by the unusual inclusion complex 2.1.5THF·MeOH, we are currently exploring its reactions with a variety of metal ions found in dinuclear metalloprotein active sites.

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Supplementary Material Available: Spectroscopic data for 1, solid-state magnetic susceptibility data for 2, and tables of atomic positional and thermal parameters for 2.1.5THF·MeOH (4 pages). Ordering information is given on any current masthead page.

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Rotamer Distribution Control and Double Michael Addition for Cyclopentane Annulation with Superb Selectivity

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> Department of Applied Chemistry, Faculty of Engineering, Okayama University Tsushima, Okayama, Japan 700 Received December 27, 1988

Michael addition of organocopper reagents to α,β -unsaturated esters and subsequent intramolecular trapping of the intermediary ester enolate with an appropriately located electrophilic center (e.g., carbonyl or halide groups) constitutes efficient and reliable route to substituted cyclopentane frameworks.¹ Although the synthetic potential of this approach can be immediately recognized, methodology allowing access to related structures with the desired absolute configurations is still left to further exploration and challenge.² We disclose herein an extremely efficient entry leading to fully substituted cyclopentane derivatives $(2)^3$ from the axially dissymmetric acyclic precursor (1) via a tandem double Michael addition reaction with superb selectivity (>99% de). This approach involves a simple and promising method for controlling groundstate rotamer distribution, so that the diastereo- π -faces of carbon-carbon double bond of the enoate 1 can be differentiated.

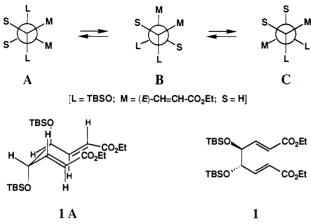
It is well known that A is the most stable rotamer among rotamers A, B, and C for the rotational equilibrium of a d,l-ethane

(3) For a carbohydrate approach to a fully substituted cyclopentane framework, see: RajanBabu, T. V. J. Am. Chem. Soc. 1987, 109, 609-610.

^{(1) (}a) Cooke, M. P., Jr. Tetrahedron Lett. 1979, 2199-2202. (b) Little, (1) (a) Cooke, M. F., Jr. *Tetraneuron Lett.* 1577, 2137-2202. (b) Little,
R. D.; Dawson, J. R. *Ibid.* 1980, 2609-2612. (c) Little, R. D.; Verhe, R.;
Monte, W. T.; Nugent, S.; Dawson, J. R. J. Org. Chem. 1982, 47, 362-364.
(d) Nugent, S. T.; Baizer, M. M.; Little, R. D. *Tetrahedron Lett.* 1982, 23, 1339-1342. (e) Nugent, W. A.; Hobbs, F. W., Jr. J. Org. Chem. 1983, 48, 254 5364-5366. See, also: Org. Synth. 1987, 66, 52-59. (f) Crimmins, M. T.; Mascarella, S. W.; DeLoach, J. A. J. Org. Chem. 1984, 49, 3033-3035. (g) Yamaguchi, M.; Tsukamoto, M.; Hirao, I. Tetrahedron Lett. 1985, 26, 1723-1726.

⁽²⁾ For an approach to optically active cyclopentane frameworks by asymmetric synthesis relying on another method, see: (a) Danheiser, R. L.; Carini, D. J.; Basak, A. J. Am. Chem. Soc. 1981, 103, 1604–1606. (b) Winterfeldt, E.; Harre, M. Chem. Ber. 1982, 115, 1437–1447. (c) Danheiser, R. L.; Carini, D. J.; Fink, D. M.; Basak, A. Tetrahedron 1983, 39, 935–947. See, also: Danheiser, R. L.; Fink, D. M.; Tsai, Y.-M. Org. Synth. 1987, 66, 8-13. (d) Misumi, A.; Iwanaga, K.; Furuta, K.; Yamamoto, H. J. Am. Chem. Soc. 1985, 107, 3343-3345.

derivative.⁴ The relative stability of the rotamer A should be



increased if L becomes so large⁵ that the rate of interconversion between A and others may become slow enough to be neglected. Diethyl (4S,5S)-4,5-bis(*tert*-butyldimethylsilyloxy)-(2E,6E)-octadienedioate (1), prepared from L-tartaric acid,⁶ can exist as the conformer 1A even at room temperature,⁷ in which the α,β -unsaturated ester moieties are gauche to each other but may try to arrange themselves in space to minimize nonbonded steric and/or scalar interactions. If realized, nucleophilic attack at the β -carbon of the enoate moiety of 1A could occur only toward the two π -faces which are exposed to the outside of the molecule. More importantly, these faces are *homotopic*.

On the basis of this working hypothesis, we have examined the Michael addition of several Grignard reagent-cuprous iodide (1:1) complexes⁸ to 1 (ether/-20 °C/1 h), and the results are summarized in Table I. In line with expectation, only one enantiomer (2a-d) (eq 1) has been obtained in every case examined.

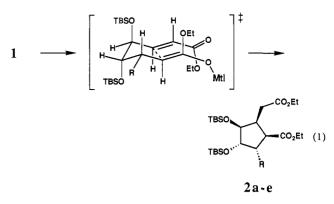
Carbon-carbon and carbon-hydrogen connectivities in the products were unambiguously determined by two-dimensional NMR spectroscopy (COSY and HETCOR), and the absolute structure confirmation was made by observing the NOE between every vicinal pair of ring protons located cis to each other.⁹ The stereochemical outcome clearly indicates that the intermolecular attack by the RMgBr-CuI complex occurred toward the π -face

(4) Eliel, E. L. Stereochemistry of Carbon Compounds; McGraw-Hill; New York, 1962; pp 124-156.

(6) Prepared from the corresponding 4,5-O-isopropylidene derivative (10) via deprotection (2N-HCl/EtOH/40 °C) and reprotection (tert-butyldimethylsilyl triflate/NEt₃/CH₂Cl₂). For the preparation of 10, see: Saito, S.; Hamano, S.; Moriyama, H.; Okada, K.; Moriwake, T. Tetrahedron Lett. 1988, 29, 1157-1160.

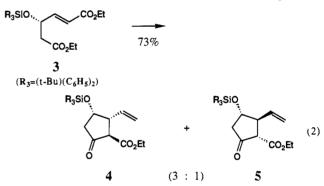
(7) McCullough, J. D. J. Am. Chem. Soc. 1940, 62, 480-484. d,l-Stilbene dibromide has shown to crystallize in the A form as verified by X-ray diffraction study.

(9) Decoupler offset cycle technique was employed with an appropriate irradiation power as weak as possible, while the power supply was gated.



exposed to the outside of the molecule, while the inside face of the remaining enoate part was attacked in the ensuing intramolecular Michael step, strongly supporting that the ground-state conformation of 1 should be that illustrated as 1A.

The amazingly high selectivity observed for the present double Michael process should be ascribed not only to the steric hindrance imposed by the γ -O-TBDMS group but also to the π -topological reason¹⁰ as mentioned above. In fact the ability of such a group to differentiate neighboring π -faces¹¹ of an α,β -unsaturated ester has been estimated to be about 3:1 based on the product distribution (4:5) observed for a tandem Michael-Dieckmann pathway (conducted under the same conditions as that for the double Michael) for the related structure (3)¹² as shown in eq 2.



This conceptually new methodology for π -face differentiation through rotamer distribution control has proven promising for other double Michael reactions mediated by heteroatom nucleophiles such as thiolate and amine. Lithium benzenethiolate has been found to react with 1 to give exclusively the corresponding adduct [2e, R = C₆H₅S, $[\alpha]^{24}_{D}$ -38.8° (c 0.99, CHCl₃)] in nearly quantitative yield.¹³ On the other hand, benzylamine failed to react with 1 even at elevated temperature and for prolonged reaction times. However, upon switching the TBDMS group to a less bulky trimethylsilyl group (6), the reaction was effected to furnish pyrrolidine nucleus (7) as a single isomer (EtOH/80 °C/48 h; 57% yield), which, on exposure to silica gel suspended in hexane, led to unstable monolactone (8a), with no trace of

⁽⁵⁾ In the case of 1,1,2,2-tetrabromoethane, a type A rotamer (L = M = Br) has been proven to be more stable than a type B rotamer (L = M = Br). It was proposed that such a relative stability in the type A rotamer stemmed from a mode of the Br-C-Br bond angle deformation (spreading out), whereby, in the B type rotamer, the bromine atom at C(1) (= M) and that at C(2) (= M) are forced to come close to each other which is also to be the case between L's (= Br's), whereas, in the A type rotamer, the gauche Br's (two M's) recede from each other. For this discussion, see: Miyagawa, I.; Chiba, T.; Ikeda, S.; Morrino, Y. Bull. Chem. Soc. Jpn. 1957, 30, 218-222. See, also: Eliel, E. L.; Allinger, N. L.; Angyal, S. J.; Morrison, G. A. Conformational Analysis, Interscience: New York, 1965; pp 18-19.

^{(8) (}a) Gilman, H.; Straley, J. M. Rec. Trav. Chim. 1936, 55, 821-834.
(b) House, H. O.; Respess, W. L.; Whitesides, G. M. J. Org. Chem. 1966, 31, 3128-3141.
(c) Posner, G. H. An Introduction to Synthesis Using Organocopper Reagents; Wiley: New York, 1980. Org. React. 1972, 19, 1-113; 1975, 22, 253-400. Excessive reagent (6-mol equiv) was required; smaller amounts of the reagent (1-5 mol equiv) resulted in almost quantitative recovery of 1. RMgBr-CuI (catalytic) initiated the reaction but was unsatisfactory in terms of the chemical conversion. Other typical organocopper reagents such as LiR₂Cu, RMgBr-CuI(catalytic)-trimethylchlorosilane (Nakamura, E.; Kuwajima, I. J. Am. Chem. Soc. 1984, 106, 3368-3370), or RLi-CuI (1:1) were totally unsuccessful. The higher order cuprate R₂CuCNLi₂ (Lipschutz, B. H. Synthesis 1987, 325-341) which was not successful for 3 and organocopper-Lewis acid combinations have not been examined (Yamamoto, Y. Angew. Chem., Int. Ed. Engl. 1986, 25, 947-959).

⁽¹⁰⁾ Asymmetric induction for 1,4-addition in this context, see: Oppolzer, W.; Loher, H. J. Helv. Chim. Acta 1981, 64, 2808-2811. Helmchen, G.; Wegner, G. Tetrahedron Lett. 1985, 26, 6047-6050. For a review, see: Tomioka, K.; Koga, K. In Asymmetric Synthesis; Morrison, J. D., Ed.; Academic Press: New York, 1983; Vol. 2, pp 201-224.

⁽¹¹⁾ For a discussion pertinent to this problem in cyclic substrate, see: Ibuka, T.; Minakata, H.; Mitsui, Y.; Kinoshita, K.; Kawai, Y. Kimura, N. *Tetrahedron Lett.* 1980, 21, 4073-4077. Ibuka, T.; Minakata, H.; Mitsui, Y.; Kinoshita, K.; Kawai, Y. J. Chem. Soc., Chem. Commun. 1980, 1193-1194.

⁽¹²⁾ Prepared from diethyl L-malate via selective reduction of the ester α to the hydroxyl group (Saito, S.; Hasegawa, T.; Inaba, M.; Nishida, R.; Fujii, T.; Nomizu, S.; Moriwake, T. *Chem. Lett.* **1984**, 1389–1396), TBDMS protection of the primary hydroxyl group, TBDPS protection of the secondary hydroxyl group, deprotection of the primary TBDMS, Swern oxidation of thus-generated hydroxyl group, and condensation with Wadsworth-Emmons reagent.

⁽¹³⁾ For an effect of the counter cation of thiolate, see: Shono, T.; Matsumura, Y.; Kashimura, S.; Hatanaka, K. J. Am. Chem. Soc. 1979, 101, 4752-4753.

J. Am. Chem. Soc., Vol. 111, No. 12, 1989 4535

Table I. Asymmetric Double Michael Addition:^a 1 + RMgBr-CuI (1:1)

entry	R	product	yield, ^b %	$[\alpha]_{\mathrm{D}}^{c}$ (deg)	% de ^d
1	CH,=CH-	2a	94	-49.1	>99
2	CH ₃ —	2 b	92	-36.8	>99
3	Сн,СН,—	2c	77	-38.1	>99
4	C ₆ H ₅ −	2d	40	-62.1	>99

^aConducted with 6-mol equiv of the reagnet in ether at -20 °C for 1 h. ^bFor the product purified by SiO₂ column chromatography. ^cIn CHCl₃ at 25 °C. ^dDetermined by ¹H (500 MHz) and ¹³C (126 MHz) NMR spectral analysis.

bis-lactone 9 being detected.¹⁴ These findings are clearly consistent with the above-mentioned consideration for the steric course of the reaction mediated by the carbon nucleophiles.¹⁵

If we resort to the π -face differentiation tactics developed in this work, other possible electrophilic, nucleophilic, and concerted processes to 1 and related structures will be rewarded with exceptionally high selectivity, which are currently our major concern.

Acknowledgment. We thank The SC-NMR Laboratory of Okayama University for high field NMR experiments (500-MHz

(14) Acetylation of 8a [4-(dimethylamino)pyridine/Ac₂O/Et₃N/CH₂Cl₂] gave the corresponding stable acetate 8b, the structure of which was confirmed by ¹H NMR as mentioned above and mass spectroscopy.

(15) Determination of the actual conformation for 1 must await clear-cut physical evidence. Nevertheless, all the results witnessed in this study seem to make the type B rotamer still less likely as a reacting form.

Book Reviews*

Phase Equilibria in Binary Halldes. By V. I. Posypaiko and E. A. Alekseeva. Compiled and edited by H. B. Bell (University of Strathclyde). Translated from Russian by B. Indyk. IFI/Plenum Data Company: New York. 1987. xxv + 470 pp. \$38.00. ISBN 0-306-65211-0.

This book is a recast translation of a Russian three-volume collection of detailed information on binary molten-salt phase diagrams. The format is condensed from the original by presenting data on the invariant points, methods of study, and reference numbers in tabular form. In addition, only the halide phase diagrams from the original are included. Detailed figures are given for 287 of the approximately 1200 phase diagrams covered and detailed tabular data are given for only a small number of systems. The 1628, largely Russian, references span the period from 1867 to 1975. No critical assessment of multiple and inconsistent data sets was attempted, and it is left to the reader to choose between different liquidus temperatures and melting points in such cases.

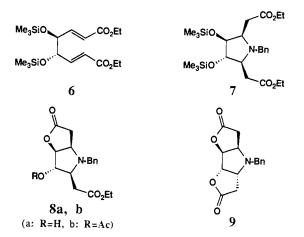
This volume presents a wealth of valuable data in a relatively compact easy-to-read format. Some of these data are difficult to find outside of the U.S.S.R. The tabular form has the advantage of presenting data more accurately than can be read from a phase diagram, but it also has the disadvantage of giving less detail. However, detail can be found in the original references, when available. The editing appears to be good. There are some slips however with, for example, one reference by Thoma appearing three times in the reference list and a small number of compound entries that have the wrong stoichiometries.

This book should be a valuable desktop reference for those working with molten salts and a desirable reference in technical libraries where high-temperature chemistry and pyrometallurgy are significant pursuits. Milton Blander, Argonne National Laboratory

Major Industrial Hazards: Their Appraisal and Control. By John Withers (Loughborough University). John Wiley and Sons: New York. 1988. xv + 241 pp. \$57.95. ISBN 0-470-21067-2.

Recently there have been a number of publications that have addressed hazardous materials. In this new work, this topic is explored in a concise but complete and detailed manner.

Major Industrial Hazards can be broken down into three parts: risk, assessment/measures, and precautions. Risk is analyzed in terms of



for ¹H and 126-MHz for ¹³C). The partial financial support by a Grant-in-Aid for Scientific Research from the Ministry of Education, Science, and Culture, Japan (63303009), is gratefully acknowledged. We deeply appreciate Dr. K. Tamao (Kyoto University) for the valuable discussions and encouragement.

Supplementary Material Available: Synthesis, spectral data (¹H NMR, ¹³C NMR, IR, and HRMS), and copies of representative NMR (¹H and ¹³C) spectra for 1, 2a, and 8b including typical COSY, HETCOR, and NOE difference spectra (18 pages). Ordering information is given on any current masthead page.

individual risk, public perception of risk, and acceptable risk. The major industrial risks are classified and specific case histories are presented. Explosive, fire, toxic, and nuclear hazards and waste disposal problems are examined. Public policy and legislation are discussed, but the scope is limited to the United Kingdom's system. An excellent chapter examines the management of industrial hazards, placing into perspective risk and the needs for proper controls.

The second part of the text explores assessment and measures of risk. In these chapters, the quantification of a release through different models proposed by Davenport, Kletz, and Fawcett is discussed. Cremer and Warner studies are described and results are given. Next, quantification of dispersion is investigated and specific materials such as LNG, propane, ammonia, chlorine, butane, and hydrogen fluoride are referred to because of their unique properties and large volumes of shipment. A very interesting chapter examines the chances of fire and explosion, and the subsequent chapter explores the damages from the fire and radiant heat, nuclear radiation, and various toxicological concerns. The final chapter in this part deals with the assessment of risk impact on a local population. Here Withers shows the parameters of various methodologies, timing, population density, and population composition.

The last segment of the text is what I believe sets it apart from other books in the field of hazardous materials and is by far this text's strength. The final three chapters deal with transportation risks, mitigation of hazards, and the costs/benefits of risk prevention. Other texts leave the topic of hazard evaluation after providing the reader with only an introduction of the controls necessary for mitigation. Withers goes into design and construction procedures of packaging, maintenance, education and training, and emergency plans to mitigate the hazards. But he then explores the risk associated with hazards that have a high probability of affecting the most people, for example, during a public-transportation accident. He has also provided the cost/benefit analysis for employees, the cost of saving a life, and the benefits to society of doing this analysis correctly.

This book would be an excellent addition to the library of any hazardous materials specialists, laboratory technicians, and operators of installations involved with processing and transportation of hazardous materials. The text is written in a style that managers would find very effective in furthering their understanding of the concepts of risk, means of mitigating the risks of handling hazardous materials, and the benefits

^{*}Unsigned book reviews are by the Book Review Editor.