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(3) Evidence for a related process involving migration of a phenyl group to electron-deficient oxygen has been reported: Reichle, W. T.; Carrick, W. L. *J. Organomet. Chem.* **1970**, *24*, 419–426. See also the reaction of didecylmercury with chromyl chloride in ref 1.

(4) For the preparation of starting materials see: Nugent, W. A.; Harlow, R. L., *Inorg. Chem.*, in press.

(5) Additional Ia could be obtained by cooling the solution but was contaminated with white needles of ^{t}BuOZnMe.

(6) Ia: mp 131 °C dec; ^{1}H NMR (C_7D_8) δ 0.98 (s, 12 H, Me), 1.40 (s, 36 H, ^{t}Bu) [methyl resonance exhibited characteristic satellites due to 14.4% ^{183}W (S = ½), J = 7 Hz]. Anal. Calcd for $C_{20}H_{48}N_4W_2$: C, 33.72; H, 6.79; N, 7.86. Found: C, 33.89; H, 6.78; N, 7.83. Ib: mp 102 °C dec; ^{1}H NMR (C_7D_8) δ 1.02 (s, 12 H, Me), 1.39 (s, 36 H, ^{t}Bu). Anal. Calcd for $C_{20}H_{48}N_4Mo_2$: C, 44.78; H, 9.02; N, 10.44. Found: C, 45.16; H, 8.81; N, 10.93.

(7) For instance, on adding 1 equiv of pyridine to Ib, the methyl resonance was shifted to δ 0.59 and the *tert*-butyl resonance to 1.48; cf. previous note.

(8) Crystal Data: $C_{20}H_{48}Mo_2N_4$; M 536.52; monoclinic; space group $P2_1/n$; at −95 °C, a = 12.941 (4), b = 9.487 (2), c = 11.226 (2) Å; β = 103.36 (2)°; Z = 2; D_c = 1.329 g cm^{-3}; Mo Kα radiation (λ = 0.71069 μ; μ = 5.1 cm^{-1}). Data were collected on a Syntex P3 diffractometer ($4° < 2\theta < 55°$) *using the ω-scan technique. The structure was solved by the Patterson* heavy-atom method and refined by the full-matrix least-squares technique. The refinement of 214 variables (anisotropic thermal parameters for Mo, N, and C, isotropic thermal parameters for H) using 2494 reflections for which $I > 2.0\sigma(I)$ yielded a conventional R index of 0.024. The largest peak in the final difference Fourier (0.37 eÅ^{-3}) was close to the Mo atom.

(9) (a) Additional important bond lengths are Mo–C(1), 2.177 (2); Mo–C(2) 2.181 (2); N(1)–C(11), 1.457 (3); N(2)–C(21), 1.487 (2); C(11)–C(av), 1.518 (4); C(21)–C(av), 1.526 (3) Å. Additional important bond angles are N(1)–Mo–N(2), 108.61 (7); N(1)–Mo–N(2)′, 166.90 (6); N(1)–Mo–C(1), 92.17 (9); N(1)–Mo–C(2), 92.37 (8); N(2)–Mo–N(2)′, 84.48 (6); N(2)–Mo–C(1), 110.44 (9); N(2)–Mo–C(2), 110.11 (8); N(2)′–Mo–C(1), 82.47 (8); N(2)′–Mo–C(2), 83.24 (8); C(1)–Mo–C(2), 135.21 (10); Mo–N(2)–Mo′, 95.52 (6); Mo–N(2)–C(21), 134.7 (1); Mo′–N(2)–C(21), 129.8 (1); N(1)–C(11)–C(av), 108.7 (2); C–C(11)–C(av), 110.2 (3); N–C(21)–C(av), 109.5 (2); C–C(21)–C(av), 109.5 (2)°. (b) A referee has inquired about the relatively large thermal ellipsoid of C(14). This effect possibly reflects the absence of any close intermolecular contacts with C(14). In contrast, C(13) has a C–H contact of 3.07 Å and C(12) has two contacts of 3.17 Å each. However, the C(11)–C(methyl) distances are all in the range 1.513 (4)–1.526 (4) Å with identical esd's.

(10) Nugent, W. A.; Haymore, B. L. *Coord. Chem. Rev.* **1980**, *31*, 123–175.

(11) The remaining organic products after hydrolysis in each case were benzene, *tert*-butylamine, trimethylsilanol, and hexamethyldisiloxane. These were identified, as were biphenyl and *N*-*tert*-butylaniline, by GC–mass spectroscopy. Authentic *N*-*tert*-butylaniline was purified by spinning-band distillation (119 °C, 45 Torr) after preparation according to Hickenbottom, W. J. *J. Chem. Soc.* **1933**, 946–951.

(12) II represents the simplest formulation for a species in which a trimethylsiloxo ligand has been replaced by a phenyl group. We cannot at this time exclude other formulations such as "ate" complexes or binuclear (Cr, Cr, or Cr, Zn) species. We are continuing our investigation into the mechanism of this reaction.

(13) Brominolysis studies appear to confirm the greater stability of the phenylmolybdenum species derived from Ib. Thus 1 mmol of Ib in hexane was treated with 2 mmol of diphenylzinc. After the resultant $PhZnOSiMe_3$ was filtered off (0.78 mmol recovered), addition of excess bromine afforded 1.5 mmol of bromobenzene, determined by GLC.

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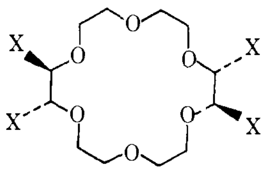
Photophysical Investigations of Chiral Recognition in Crown Ethers

Sir:

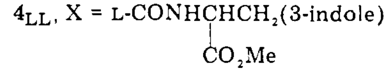
The present communication reports the first use of photophysical techniques for the elucidation of chiral recognition in crown ethers.[1-10] Fluorescence quenching, energy transfer, and excimer formation have provided information on the complexation of achiral and chiral guests, on the conformation of functionalized crown ethers and on the effects of solvents and guests on given conformations.

1,2,10,11-(*S*,*S*,*S*,*S*)-(−)-Tetracarbo(*N*,*N*)dimethylamido-3,6,9,12,15,18-hexaoxocyclooctadecane (L-crown-amide, $\mathbf{1}_{\mathrm{L}}$) or its enantiomer, 1,2,10,11-(*R*,*R*,*R*,*R*)-(+)-tetracarbo-(*N*,*N*)dimethylamide-3,6,9,12,15,18-hexaoxocyclooctadecane (D-crown-amide, $\mathbf{1}_{\mathrm{D}}$),[11] were used as starting materials for the synthesis of optically active fluorescent crown ethers. $\mathbf{1}_{\mathrm{L}}$ and $\mathbf{1}_{\mathrm{D}}$ were converted into their acids, $\mathbf{2}_{\mathrm{L}}$ and $\mathbf{2}_{\mathrm{D}}$. The acids provided the acid chlorides, $\mathbf{3}_{\mathrm{L}}$ and $\mathbf{3}_{\mathrm{D}}$, which, in turn, allowed the preparation of L-crown-D-Trp ($\mathbf{4}_{\mathrm{LD}}$), L-crown-L-Trp ($\mathbf{4}_{\mathrm{LL}}$), D-crown-D-Trp ($\mathbf{4}_{\mathrm{DD}}$), D-crown-L-Trp ($\mathbf{4}_{\mathrm{DL}}$),[12] L-crown-pyr ($\mathbf{5}_{\mathrm{L}}$), and D-crown-pyr ($\mathbf{5}_{\mathrm{D}}$).[13]



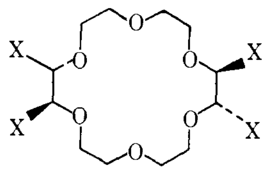
$\mathbf{1}_{\mathrm{L}}$, X = $CON(CH_3)_2$
$\mathbf{2}_{\mathrm{L}}$, X = CO_2H
$\mathbf{3}_{\mathrm{L}}$, X = COCl

$\mathbf{4}_{\mathrm{LD}}$, X = $\text{D-CONH}\underset{\text{CO}_2\text{Me}}{\underset{|}{\text{C}}}\text{HCH}_2\text{(3-indole)}$

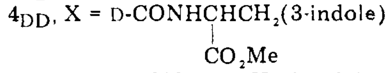


$\mathbf{4}_{\mathrm{LL}}$, X = $\text{L-CONH}\underset{\text{CO}_2\text{Me}}{\underset{|}{\text{C}}}\text{HCH}_2\text{(3-indole)}$

$\mathbf{5}_{\mathrm{L}}$, X = CONH-1-pyrene



$\mathbf{1}_{\mathrm{D}}$, X = $CON(CH_3)_2$
$\mathbf{2}_{\mathrm{D}}$, X = CO_2H
$\mathbf{3}_{\mathrm{D}}$, X = COCl



$\mathbf{4}_{\mathrm{DD}}$, X = $\text{D-CONH}\underset{\text{CO}_2\text{Me}}{\underset{|}{\text{C}}}\text{HCH}_2\text{(3-indole)}$

$\mathbf{4}_{\mathrm{DL}}$, X = $\text{L-CONH}\underset{\text{CO}_2\text{Me}}{\underset{|}{\text{C}}}\text{HCH}_2\text{(3-indole)}$

$\mathbf{5}_{\mathrm{D}}$, X = CONH-1-pyrene

Absorption spectra of all isomeric crown ether tryptophans were identical in methanol.[14] Small, but noticeable, differences were observed, however, between the emission intensities of the diastereomers: $\Phi_{4_{\mathrm{DL}}} = 0.200 \pm 0.008$, $\Phi_{4_{\mathrm{LL}}} = 0.180 \pm 0.008$.[15] Addition of $TbCl_3$ decreased the fluorescence yields. Stern-Volmer plots for 5.0×10^{-5} M D-crown-L-Trp and L-crown-L-Trp in MeOH were linear up to $\sim 5.0 \times 10^{-5}$ M $TbCl_3$, after which they leveled off. This data implies the quenching of the excited states by Tb^{3+} ions bound in the cavities of the crown ethers. Treatment of fluorescence intensities in the absence and in the presence of different amounts of $TbCl_3$[16] leads to binding constants of K(Tb^{3+}/D-crown-L-Trp) = $(2.90 \pm 0.21) \times 10^4$ M^{-1} and K(Tb^{3+}/L-crown-L-Trp) = $(2.76 \pm 0.12) \times 10^4$ M^{-1}. These values are of the same magnitude as determined for metal ion-crown ether complexes.[17] Complexing an achiral guest into crown ethers is seen to be unaffected by the chirality of the host. Cavity diameters are apparently the same for D-crown-L-Trp and L-crown-L-Trp. Only modest chiral recognitions have been observed using glycine-L-phenylalanine (Gly-L-Phe) and glycine-D-phenylalanine (Gly-D-Phe). Treatment of fluorescence intensities of D-crown-L-Trp (5.0×10^{-5} M in MeOH) in the absence and in the presence of different amounts of chiral guests[16] leads to K(Gly-L-Phe/D-crown-L-Trp) = $(1.3 \times 0.1) \times 10^4$ M^{-1} and K(Gly-D-Phe/D-crown-L-Trp) = $(1.9 \pm 0.1) \times 10^4$ M^{-1}.

Substantial chiral discriminations are seen, however, in the efficiencies of energy transfer from the tryptophan side arms

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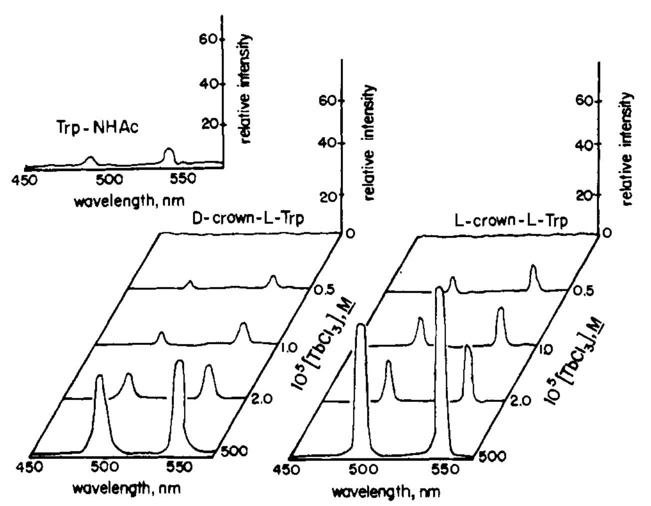

Figure 1. Relative emission intensities of 5.0×10^{-3} M $TbCl_3$ in the presence of 2.0×10^{-4} M TryNHAc (upper left corner). Three-dimensional plots of emission spectra of varying amounts of $TbCl_3$ in the presence of 5.0×10^{-5} M D-crown-L-Trp and L-crown-L-Trp (excited at 290 nm) in MeOH.

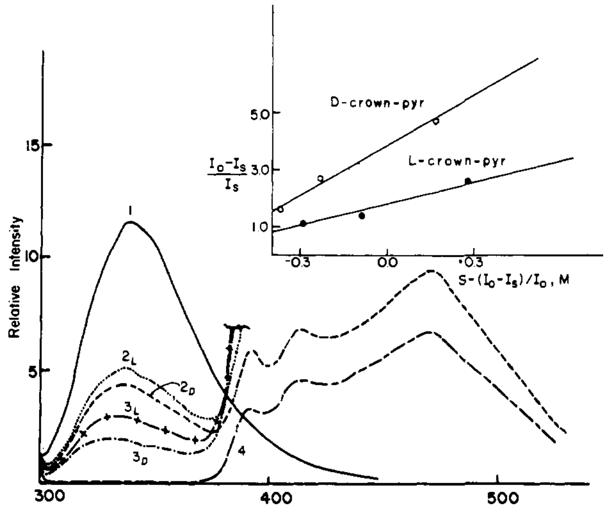

Figure 2. Emission spectra of 2.0×10^{-6} M L-crown-pyr in ethylene glycol (1, -+-), DMF (2, - - -), THF (3, —), THF saturated by KCl (stirred overnight) (4, - - -), THF containing 1.25×10^{-3} M $TbCl_3$ (5, - · -). Emission spectra of 8.0×10^{-6} M PyNHAc in THF (6, · · ·) are also included for comparison. All spectra were taken at an excitation wavelength of 334 nm and at 25 °C. Inset shows plots of quenching data according to equation given in note 21. All spectra were obtained at excitation wavelength of 290 nm.

of the crown hosts to the trapped Tb^{3+} guests in 4_{LL} and 4_{DL} (Figure 1). In the absence of crown ethers, energy transfer is negligible (see upper left corner in Figure 1). Energy transfer becomes efficient when the donors, the flexible tryptophan arms, can most closely approach the Tb^{3+} acceptors, localized in the cavities of the crown ethers. This type of energy transfer is, therefore, a sensitive measure of host conformational changes. The almost twice as efficient energy transfer in the L-crown-L-Trp/Tb^{3+} complex than that in the D-crown-L-Trp/$Tb_3{}^+$ complex implies an appreciably shorter average interchromophoric distance in the former than in the latter.

The bulky pyrene side arms in 5_L and 5_D are sufficiently close to allow for intramolecular excimer formation. Excimer to monomer ratios are highly solvent dependent (Figure 2). In alcohols (MeOH, ethylene glycol) excimer formation predominates; in CH_2Cl_2 THF[18] and DMF monomers coexist with excimers.[19] Qualitatively, fluorescence quenching by $TbCl_3$ is similar to that observed for the typtophan containing crown ethers. Hence, there is no discrenable difference in quenching, in the binding of Tb^{3+} between D-crown-pyr and L-crown-pyr. Quantitatively the situation is quite complex. Quenching the singlet manifold by a metal ion, bound in the crown ether cavity, competes with excimer formation. Concomitantly, the average pyrene-pyrene interchromophoric distances, hence the efficiency of excimer formation, may well be altered upon complexation. Alteration of monomer:excimer ratios by a *nonquenching* molecule provides, therefore, evidence for conformational changes of the host upon complexation of a guest. This phenomenon is illustrated by the effect of K^+ complexation on the emission spectra of L-crown-pyr in THF. It is seen in Figure 2 that addition of KCl (a guest which does not quench the fluorescence of pyreneacetamide) enhances the excimer at the expense of the monomer emission (compare spectra 3 and 4 in Figure 2).

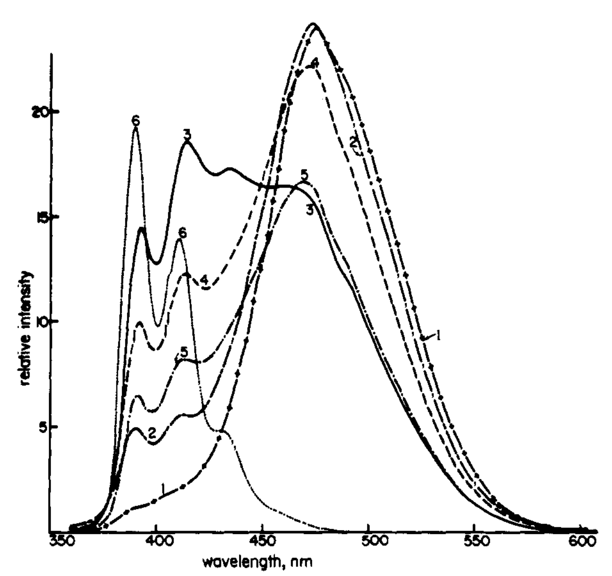

Figure 3. Emission spectra of 5.0×10^{-5} Gly-L-Trp in THF in the presence of 0 (1), 1.25×10^{-5} M D-crown-pyr (2_D), 1.25×10^{-5} M L-crown-pyr (2_L), 5.0×10^{-5} M D-crown-pyr (3_D), 5.0×10^{-5} M L-crown-pyr (3_L). For comparison, the emission spectrum of 1.25×10^{-5} M L-crown-pyr is also given in THF (4).

Fluorescence intensities of glycine-L-tryptophan (Gly-L-Trp) have been quenched differentially by D-crown-pyr and L-crown-pyr in THF (Figure 3).[20] Emission intensities of 5.0×10^{-5} M Gly-L-Trp as functions of added crown ethers lead to[21] K(Gly-L-Trp/D-crown-pyr) = $(1.2 \times 0.2) \times 10^5$ M^{-1} and K(Gly-L-Trp/L-crown-pyr) + $(5.0 \pm 0.3) \times 10^4$ M^{-1}. These binding constants are factors of 2.5 to 10 greater than those observed for the binding of Gly-L- (or D-) Phe to tryptophan-carrying crown ethers in methanol. More significantly, chiral recognition in binding is K(Gly-L-Trp/D-crown-pyr)/K(Gly-L-Trp/L-crown-pyr) = 2.4. Alternatively expressed, the free energies of binding ranged from 6.1 to 7 kcal/mol and chiral recognition from 0.23 to 0.52 kcal/mol, and therefore a "chiral efficiency" of ~4-7% was realized. This substantial chiral recognition in complexation is the consequence of the more pronounced interactions between the tryptophan moiety of the guest and the pyrene side chain of the host than those observed in tryptophan containing crown ethers.

Proper alignments of host and guest moieties have been recognized for some time as a requirement for maximizing chiral recognition in crown ethers.[1] Photophysical investigations, initiated in the present work, shows just how much different substituents on the crown ether, different guests, and solvents can influence chiral recognition. This approach allows quantitative assessments of the parameters involved and thus provides the required knowledge for the design of host-guest systems capable of maximum chiral recognition. Elucidation of kinetic and thermodynamic details of chiral recognition in the present and other systems is the subject of our current attention.

Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for their support of this research by a New Directions in Fundamental Research grant. Pietro Tundo thanks the Italian Consiglio Nazionale delle Ricerche for financial support.

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(11) Synthesis of $\mathbf{1}_{\mathrm{L}}$ and $\mathbf{1}_{\mathrm{O}}$ followed that described for $\mathbf{1}_{\mathrm{L}}$.[7] Analytical and ^{1}H NMR data are in agreement with the proposed structures and with those reported previously.[7] $\mathbf{1}_{\mathrm{L}}$ had mp 181–182 °C, $[\alpha]_{\mathrm{D}}^{25}$ +108.4° (*c* 1.5, $CHCl_3$); $\mathbf{1}_{\mathrm{O}}$ had mp 181–182 °C, $[\alpha]_{\mathrm{D}}^{25}$ −108.3°.

(12) $\mathbf{4}_{\mathrm{LL}}$, $\mathbf{4}_{\mathrm{LO}}$, $\mathbf{4}_{\mathrm{OO}}$, and $\mathbf{4}_{\mathrm{OL}}$ were prepared by adding $\mathbf{3}_{\mathrm{L}}$ or $\mathbf{3}_{\mathrm{O}}$ (0.23 mmol, 0.12 g) in THF (10 mL) dropwise to a stirred suspension of the appropriate tryptophan methyl ester HCl (2.0 mmol, 0.51 g) and triethylamine (0.5 mL) in THF (50 mL). After 2 h of stirring, the THF was removed under vacuum, CH_2Cl_2 added, and the organic layer washed with 2 N HCl, 5% $NaHCO_3$, and water (twice each). Subsequent to solvent removal and drying, crystallization from CH_2Cl_2 was initiated by ether. Following two recrystallizations, yields, melting points, and rotations were as follows: $\mathbf{4}_{\mathrm{LL}}$, 65%, mp 135–138 °C, $[\alpha]_{\mathrm{D}}^{25}$ +8.2° (*c* 0.6, MeOH); for $\mathbf{4}_{\mathrm{OL}}$, 70%, mp 145–150 °C, $[\alpha]_{\mathrm{D}}^{25}$ −3.0° (*c* 0.6, MeOH); for $\mathbf{4}_{\mathrm{OO}}$, 77%, mp 135–138 °C, $[\alpha]_{\mathrm{D}}^{25}$ −8.3° (*c* 0.6, MeOH); for $\mathbf{4}_{\mathrm{LD}}$, 70%, mp 145–150 °C, $[\alpha]_{\mathrm{D}}^{25}$ +3.0° (*c* 0.6, MeOH). Satisfactory elementary analyses were obtained for all compounds and ^{1}H NMR confirmed the proposed structures.

(13) Preparations and purifications of $\mathbf{5}_{\mathrm{L}}$ and $\mathbf{5}_{\mathrm{O}}$ followed those used for their tryptophan analogues.[12] Yields, melting points, and rotations: for $\mathbf{5}_{\mathrm{L}}$, 57%, mp 248 °C dec, $[\alpha]_{\mathrm{D}}^{25}$ +11.3° (*c* 0.6, THF); $\mathbf{5}_{\mathrm{O}}$, 65%, mp 245 °C dec, $[\alpha]_{\mathrm{D}}^{25}$ −11.2° (*c* 0.6, THF).

(14) 5.0×10^{-5} M solutions; $\epsilon_{291\ \mathrm{nm}}$ 2.2×10^4, $\epsilon_{282\ \mathrm{nm}}$ 2.6×10^4, $\epsilon_{27\mathrm{nm}\ (\mathrm{sh})}$ 2.4×10^4 M^{-1} cm^{-1} (Cary 118C spectrophotometer).

(15) Emission spectra were taken on samples degassed on a high vacuum line by repeated freeze–pump–thaw cycles using a SPEX Fluorolog instrument exciting with 290-nm light (10-nm band path). Quantum yields are related to L-TrpNHAc in methanol, taking $\varphi_{\text{L-TrpNHAc}} = 0.20$.

(16) Host–guest binding constants were calculated from the equation $(I_o - I_s)/(I_s - I_\infty) = K[S - (I_o - I_s)(I_o - I_\infty)]C$ where I_o, I_s, and I_∞ are emission intensities of the host at 350 nm (excited at 290 nm) in the absence, in the presence of different amounts, and in complete quenching by the guest, respectively; C is the concentration of the host (5.0×10^{-5} M); and S is the molar ratio of guest:host.

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(18) THF was freshly distilled each time, prior to use.

(19) Monomer to excimer ratios are also temperature dependent. In a given solvent, decreasing temperatures favor excimer formation. This, presumably, is a composite effect of conformational changes which alters the proximity of neighboring pyrenes and the free energy requirements of excimer formation.

(20) Quenching of Gly-L-Trp fluorescence is seen (in Figure 3) to be paralleled by enhanced emission of the pyrene chromophore of the crown ether. The presence of excimers precludes the elucidation of energy transfer efficiencies from the present data.

(21) Host–guest binding constants were calculated from equation $(I_o - I_s)/I_s = K[S - (I_o - I_s)/I_o]C$ where I_o and I_s are emission intensities of the guest (Gly-L-Trp) at 350 nm (excited at 290 nm) in the absence of different amounts of quenching by the host, respectively; C is the concentration of the guest (5.0×10^{-5} M); and S is the molar ratio of host:guest.

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Book Reviews

Chemical and Biochemical Applications of Lasers. Volume IV. Edited by C. BRADLEY MOORE. Academic Press Inc., New York. 1979. xi + 405 pp. $25.00.

The fourth volume in a continuing series about chemical and biochemical research using lasers, this volume is a collection of eight research articles describing different areas. The topics covered are: laser-induced fluorescence spectroscopy; resonance, surface and coherent anti-Stokes Raman spectroscopy; theory of molecular rate processes in the presence of intense laser radiation; multiphoton dissociation of gas-phase ions using low intensity laser radiation; and photochemical fixation of nucleic acid double helix utilizing psoralens.

There is a good mix of theory and experiment and the contributors have made a visible effort to present their results in simple terms, but it is still for those who already know the fundamentals of laser spectroscopy and not for the laymen. After reading through the book, one point clearly comes across: that there is still enormous potential for lasers in new areas of research. As a whole, it is a valuable addition to the large laser-research literature.

Erdogan Gulari, *The University of Michigan*

Contemporary Topics in Polymer Science. Volume 1. Macromolecular Science Retrospect and Prospect. Edited by R. D. ULRICH. Plenum Press, New York. 1978. ix + 284 pp. $29.50.

This volume comprises the proceedings in honor of the 25th anniversary of the Division of Polymer Chemistry of the ACS, which occurred in 1976. It includes a history of the Division, largely statistical and tabular, contributed by the Editor. The remainder of the volume contains retrospective comments by some sixteen prominent polymer chemists on their particular contributions. The chemists represented here are: F. A. Bovey, F. R. Eirich, J. D. Ferry, P. J. Flory, M. L. Huggins, L. Mandelkern, H. F. Mark, C. S. Marvel, B. Maxwell, H. Morawetz, M. Morton, C. G. Overberger, A. Peterlin, C. E. Schildknecht, J. K. Stille, and M. Szwarc. The chapters, one from each contributor, range widely in style. A few may be read as short introductions to a particular topic, while others tersely summarize the contributor's research field. Historical notes on places and institutions, and anecdotal material, are scattered throughout the text. Several of the authors include a complete list of their publications, while several others discuss their plans for future research.

George D. J. Phillies, *The University of Michigan*

Electronic Structure and Magnetism of Inorganic Compounds. Volume 5. P. DAY, Senior Reporter (University of Oxford). The Chemical Society, London. 1977. viii + 248 pp. £21.00.

Volume 5 of "Electronic Structure and Magnetism of Inorganic Compounds" discusses advances in the field during 1974 and 1975. Nearly 1600 references are cited in the four chapters: Electronic Spectra, by P. Day; Magnetic and Natural Optical Activity, by A. J. McCaffery; Magnetic Susceptibility Measurements, by A. K. Gregson; and Luminescence Properties of Inorganic Compounds, by D. J. Robbins and A. J. Thomson. Unfortunately, a planned fifth chapter on photoelectron spectroscopy could not be included.

The authors have produced a critical survey of research in the four areas, emphasizing the most significant advances in each. The report is not quite exhaustive; the more routine work in some instances has been sifted out. What remains is a treasury of experimental and theoretical results, tied together with expert commentary. Although the reports in this series are written for the "active specialist chemist", they will certainly be found valuable by the interested nonspecialist as well. As financially pinched libraries continue to cut back on their journal subscriptions, researchers will be forced to depend more heavily on periodical reports such as these. It can only be hoped that the high quality of this series is maintained in subsequent volumes.

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