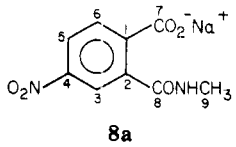
8b, or *N,N*-dimethylbenzamide) given]: 1, 145.4 (145.7); 2, 138.5 (133.0); 3, 122.4 (124.2); 4, 146.9 (148.8); 5, 120.8 (123.9); 6, 130.8 (130.0); 7, 170.5 (170.4); 8, 166.7 (166.5); 9 and 10, 34.2 and 37.9 (34.5 and 38.6); 1′, 143.9 (143.3); 2′, 140.6 (139.6); 3′, 123.2 (121.6); 4′, 146.7 (147.5); 5′, 124.4 (123.6); 6′, 127.3 (129.6); 7′, 171.1 (169); 8′, 165.9 (168); 9′ and 10′, 34.2 and 37.9 (34.5 and 38.6).

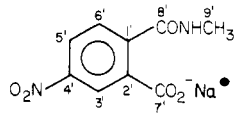
(Assignments for C-3 and C-5 might be switched; also those of C-3′ and C-5′ might be switched.)

Attempted Reaction of the Mixture of 10a and 10b with 2 and K_2CO_3 in Me_2SO. Exactly 1.19 g (0.005 mol) of the mixture of **9a** and **9b** (isolated above) and 0.54 g (0.005 mol) of **2** in 15 mL of Me_2SO were stirred with 3.45 g (0.025 mol) of K_2CO_3 at 25 °C for 10 min (CO_2 gas evolution) and then at 142 °C for 3.5 h under N_2. Analysis of the resulting supernatant solution indicated that only **10a**, **10b**, and **2** were present. No detectable nitro displacement had occurred. For **2**, the ^{13}C chemical shifts were 20.16, 115.2, 126.8, 129.6, and 155.5, all identical with those for authentic **2**;[11] for **10a**: 145.3, 138.6, 122.5, 147.1, 120.9, 130.8, 170.4, 166.1, 34.2, 37.9; for **10b**: 144.0, 140.2, 123.4, 146.8, 124.4, 127.4, 171.0, 166.1, 34.2, 37.9. The mole ratio determined for **10a**:**10b** in this spectrum was 26:74.

Preparation of the Mixture of Sodium Salts of 6a and 6b. A mixture of 20.62 g (0.100 mol) of **1**, 12 mL of H_2O, and 7.90 g of 50.62% NaOH (0.100 mol of NaOH) was stirred and heated at 45–50 °C for 40 min. (The system had become homogeneous after ca. 10 min.) Complete water removal (as determined by ^1H NMR) in vacuo at ca. 80 °C gave 24.3 g (99%) of **8a**(Na^+) + **8b**(Na^+) as a yellow powder. Structural confirmation was obtained by ^{13}C NMR (Me_2SO-d_6) spectroscopy [carbon atom (**8a** and **8b**),



8a



8b

chemical shift given]: 1, 146.3; 2, 135.1; 3, 124.2; 4, 147.5; 5, 123.3; 6, 130.2; 7, 171.4; 8, 168.1; 9 and 9′, 26.5; 1′, 141.9; 2′, 141.2; 3′, 122.8; 4′, 147.1; 5′, 124.2; 6′, 129.4; 7′, 169.9; 8′, 169.8; mole ratio of **8a**(Na^+):**8b**(Na^+) 61:39.

Reaction of 8a(Na^+) + 8b(Na^+) with 2 in Me_2SO. A mixture of 2.46 g (0.0100 mol) of **8a**(Na^+) + **8b**(Na^+) from above and 1.08 g (0.0100 mol) of **2** in 30 mL of Me_2SO (*no* K_2CO_3) was heated at 142 °C under N_2 for 3.5 h. **2** and **3** in the mole ratio of 20:80 were readily observed by ^{13}C NMR spectroscopy of the reaction mixture, as was a small amount of **8**. High-pressure LC analysis of a sample of the reaction mixture indicated that **1**, **2**, **3**, and **8** were present in the ratio of 2.1:14.1:85.8:7.2, respectively. A minor impurity (ratio 0.7) of 4,4′-oxybis(*N*-methylphthalimide] was also detected. The high-pressure LC parameters (retention time in seconds, $\epsilon^{254}/\epsilon^{280}$, and the area to moles conversion factors) were as follows: 1: 266, 5.78, 2.50; **2**: 232, 0.12, 4.41; **3**: 534, 5.26, 2.27; **8**: 82, 1.0, 0.88; the bisimide impurity: 391, 6.32, 1.52.

Registry No. 1, 41663-84-7; **2**, 106-44-5; **3**, 72709-41-2; **4a**, 72709-42-3; **4a**, potassium salt, 72709-43-4; **4b**, 72709-44-5; **4b**, potassium salt, 72709-45-6; **5**, 63196-15-6; **5**, dipotassium salt, 72709-46-7; **6a**, 72709-47-8; **6a**, sodium salt, 72709-48-9; **6b**, 72709-49-0; **6b**, sodium salt, 72709-50-3; **8a**, 72709-51-4; **8b**, 72709-52-5; **9a**, 72709-53-6; **9a**, potassium salt, 72709-54-7; **9b**, 72709-55-8; **9b**, potassium salt, 72709-56-9; **11**, 63196-09-8; **12**, 63196-28-1; **13**, 72709-57-0; 2-methyl-1*H*-isoindole-1,3(2*H*)-dione, 550-44-7; 1,2-benzenedicarboxylic acid, 88-99-3; 1,3-isobenzofurandione, 85-44-9; 2-phenyl-1*H*-isoindole-1,3(2*H*)-dione, 520-03-6; sodium 4-methylphenoxide, 106-44-5; 4-nitrophthalic anhydride, 5466-84-2; dimethylamine, 124-40-3.

Supplementary Material Available: A substantial amount of the Experimental Section, methods used for calculating chemical shifts, the method used to calculate the ^{13}C NMR substituent effects of a 4-methylphenoxy group, and accompanying tables of data (25 pages). Ordering information is given on any current masthead page.

(14) L. M. Jackman, "Applications of Nuclear Magnetic Resonance Spectroscopy in Organic Chemistry", Pergamon Press, New York, 1959, p 85.

(15) The low yield and low relative proportion of **6b** suggest that this isomer was preferentially lost in the workup procedure used.

Use of Liquid-Crystal-Induced Circular Dichroism for Determination of Absolute Configuration of Alcohols and Oxaziridines

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Addition of small amounts of chiral dopants to the achiral nematic liquid crystal, *N*-*p*-methoxybenzylidene-*p*'-*n*-butylaniline (MBBA), gives rise to liquid-crystal-induced circular dichroism (LCICD). For a variety of secondary alcohol dopants, it is shown that the sign of the LCICD band can be correlated with the absolute configuration of the dopant. The reasons underlying this phenomenon are discussed and the technique is extended to assign absolute configurations to two optically active oxaziridines.

Introduction

Recent studies of liquid crystals have provided the basis for an interesting and potentially useful technique for assigning absolute configuration.[1-5] This technique, liquid-crystal-induced circular dichroism (LCICD),[1] is extremely sensitive and can be considered as a method of chirality amplification.

Achiral molecules exhibit chiroptic behavior during association with a chiral medium.[1] Conversely, an achiral medium can assume chiroptic properties upon addition of a chiral substance.[2,4,5] For example, addition of a small amount of a chiral dopant to an achiral nematic liquid crystal such as *N*-*p*-methoxybenzylidene-*p*'-*n*-butylaniline (MBBA) causes the latter to become cholesteric and to show strong CD activity for the MBBA chromophore. The sign of the CD band depends upon the helical sense of the mesophase and is independent of the presence or absence of chromophores in the chiral dopant. This technique has been proposed as a basis for determining the absolute configuration of the chiral dopant[4] and is especially at-

(1) F. D. Saeva and J. J. Wysocki, *J. Am. Chem. Soc.*, **93**, 5928 (1971).
(2) F. D. Saeva, *Mol. Cryst. Liq. Cryst.*, **23**, 171 (1973).
(3) G. W. Gray and D. G. McDonnel, *Mol. Cryst. Liq. Cryst.*, **34**, 211 (1977).
(4) (a) G. Gottarelli, B. Samori, S. Marzocchi, and C. Stremmenos, *Tetrahedron Lett.*, 1981 (1975); (b) G. Gottarelli, B. Samori, U. Folli, and G. Torre, *J. Phys. Colloq.*, C3-25 (1979).
(5) (a) E. H. Korte, B. Schrader, and S. Bralek, *J. Chem. Res. (S)*, 236, (M) 3001 (1978); (b) B. J. Krabbe, H. Hezzemeier, B. Schrader, and K. H. Korte, *J. Chem. Res. (S)* 238, (M) 3023 (1978).

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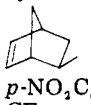
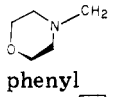
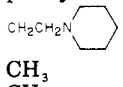
Table I. LCICD of Some Chiral Alcohols

R	R′	configuration[a]	induced[b] CD
CF_3	phenyl	(*S*)-(+)-**1a**	+
CF_3	α-naphthyl	(*S*)-(+)-**1b**	−
CF_3	9-anthryl	(*S*)-(+)-**1c**	−
CF_3	9-(10-methylanthryl)	(*S*)-(+)-**1d**	−
CF_3	9-(10-bromoanthryl)	(*S*)-(+)-**1e**	−
CF_3	9-(10-phenylanthryl)	(*S*)-(+)-**1f**	−
CF_3	10-(1-phenylanthryl)	(*S*)-(+)-**1g**	−
CF_3	9-(10-benzylanthryl)	(*S*)-(+)-**1h**	−
9-(10-thiomethylanthryl)	CF_3	(*R*)-(−)-**1i**	+
$CF_2CF_2CF_3$	phenyl	(*S*)-(+)-**1j**	+
CCl_3	phenyl	(*S*)-(+)-**1k**	+
phenyl	CH_3	(*S*)-(−)-**1l**[c]	+
$CF_2CF_2CF_3$	cyclohexyl	(*S*)-(−)-**1m**	+
$CF_2CF_2CF_3$		(*S*)-(−)-**1n**	+
CF_3	*p*-$NO_2C_6H_4$	(*S*)-(+)-**1o**	−
3,5-$(NO_2)_2C_6H_3$	CF_3	(*R*)-(−)-**1p**	+
3-pyrenyl	CF_3	(*R*)-(−)-**1q**	+
CH_3	mesityl	(*R*)-(+)-**1r**[c]	−
4-pyridyl	CH_3	(*S*)-(−)-**1s**[c]	+
3-pyridyl	CH_3	(*S*)-(−)-**1t**[c]	+
$CH_2N(CH_3)_2$	phenyl	(*S*)-(+)-**1u**[c]	−
phenyl		(*R*)-(−)-**1y**[c]	+
$CH_2CH_2N(CH_3)_2$	phenyl	(*R*)-(+)-**1w**[c]	−
phenyl		(*S*)-(−)-**1x**[c]	+
α-naphthyl	CH_3	(*S*)-(−)-**1y**[c]	+
cyclohexyl	CH_3	(*S*)-(+)-**1z**[c]	+
CH_2CH_3	CH_3	(*S*)-(+)-**1aa**[c]	+
$CH_2N(CH_3)_2$	$C(CH_3)_3$	(*S*)-(−)-**1bb**[c]	−
$CH_2N(CH_3)$	CH_3	(*S*)-(−)-**1cc**[c]	−

[a] The LCICD's of the *R* enantiomers of **1a-1g**, **1j**, **1m**, and **1n** have also been examined, and are opposite those of the *S* enantiomers. [b] At the MBBA absorbance (390-410 nm). [c] From ref 4.

tractive in view of the rather small amounts (>0.1 mg) of chiral dopant required. While the absolute configuration of a given chiral dopant clearly determines the sense of the induced helical order and attendant LCICD, one might well ask as to the extent it will be possible to relate the sign of the LCICD to the absolute configurations and structures of an extended series of chiral dopants.

In a pioneering effort, Gottarelli et al.[4a] observed a consistent relationship between the absolute configurations of some chiral secondary alcoholic dopants and the sign of LCICD for MBBA. This work subsequently was extended to cover a series of optically active sulfoxides.[4b] Correlation of the sign of the LCICD with the absolute configuration of the sulfoxide required assessment of the effective volume of the groups bonded to sulfur. Relative "effective volumes" appear to depend upon the nematic matrix employed.[4b] An interesting variation employing infrared LCICD for correlation of the absolute configurations of an assortment of solute types has been the object of several recent reports.[5] We herein provide a number of additional examples of LCICD, show that the phenomenon also extends to oxaziridine dopants, comment upon the possible origin of the secondary alcohol induced phenomenon, and utilize the technique to empirically assign the absolute configurations of two oxaziridines.

Results and Discussion

Alcohols. The LCICD observed for a number of chiral secondary alcohols dissolved in MBBA (a room temperature nematic liquid crystal) are presented in Table I. Circular dichroism can be observed for the MBBA chromophore (390–410 nm) at room temperature, a positive band indicating right-handed helical order in the mesophase. No pitch band could be observed between 200 and 700 nm and presumably occurs in the infrared. Except for compound **1cc** (last entry, Table I) the sign of the induced CD can be correlated with the absolute configuration of the dopant as determined from a Cahn–Ingold–Prelog-like system with a substituent priority scheme based upon repulsive ability (similar but not identical to size) of the substituents.

Gottarelli et al.[6] have previously postulated a mechanism to account for the observed LCICD of alcohols **1q**–**1cc**. It was noted (infrared) that alcohols hydrogen bond to the imine nitrogen of MBBA with the OH bond approximately perpendicular to the long axis of MBBA. Gottarelli also points out that monosubstituted benzenes are known to have a slight preference for an orientation in which the long axis is aligned with the nematic director and the plane of the benzene ring is parallel to that of the aryl substituents of the MBBA molecules. Hence, it was concluded that **1** aligns in MBBA as shown in Figure 1. As a consequence, MBBA molecules in adjacent layers were presumed to skew to avoid interaction with the remaining bulky substituent of **1**. This skew gives rise to the helical order and

(6) G. Gottarelli, B. Samori, and C. Stremmenos, *Chem. Phys. Lett.*, 40, 308 (1976).

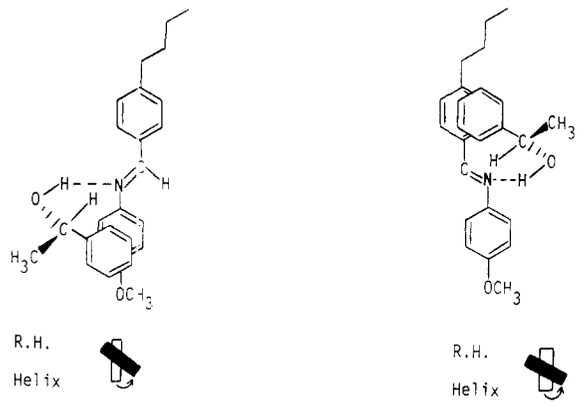


Figure 1. Alignment of (*S*)-1-phenylethanol (**11**) in MBBA proposed by Gottarelli et al.[6] The hydroxyl proton of **11** hydrogen bonds to the lone pair of electrons on nitrogen, and the aryl ring of **11** overlaps with that of MBBA. The MBBA molecule above this complex skews to avoid interaction with the methyl group, thus forming a segment of a right-handed helix.

determines the sign of the LCICD. Nonaromatic secondary alcohols could be fitted to this model by assuming that the largest or most planar substituent played the former intercalation role of the aromatic substituent.

Having long been concerned with the details of solvation by secondary alcohols as well as instrumental methods for determining enantiomeric purity and absolute configuration, we were intrigued not only by the obvious practical applications of LCICD but also by the possibility that secondary alcohol induced LCICD might have its origin in specific solvation modes reminiscent of those we had previously studied in NMR and high-performance LC.

From Table I, it may be seen that portions of our data for secondary alcohols (i.e., **1j**, **1k**, **1m**, and **1n**) do not conform to Gottarelli's model. It is possible, however, to amend this model so that it is consistent with essentially all the data in Table I through analogy with other established intermolecular interactions.

Carbinyl hydrogen bonding has been invoked as a source of conformational control.[7] If, concomitant with hydroxyl hydrogen bonding to the lone pair of electrons on the imine nitrogen, there is carbinyl hydrogen bonding to either an aromatic ring of MBBA (it makes no difference which one) or the imine π bond, the two remaining carbinyl substituents could be disposed above and roughly to either side of the long axis of MBBA. Thus, whereas Gottarelli considers the relative sizes of the carbinyl hydrogen and the *remaining nonintercalating substituent*, the modified model considers the relative repulsive effects of the two carbinyl substituents of the secondary alcohol. That substituent least repellent (or conceivably attractive) to the adjacent layer of MBBA molecules might then determine the sense of the skew of that layer, since nearby MBBA molecules would prefer to align adjacent to this substituent. Such repulsive effects could be steric or electronic in origin. There is a body of data that suggests the existence of repulsive interactions between perfluoroalkyl groups and aryl systems well out of proportion to the size of the perfluoroalkyl group.[8] Hence, the adjacent aromatic MBBA molecules presumably skew away from perfluoroalkyl substituents even though this substituent may be smaller than the remaining carbinyl substituent.

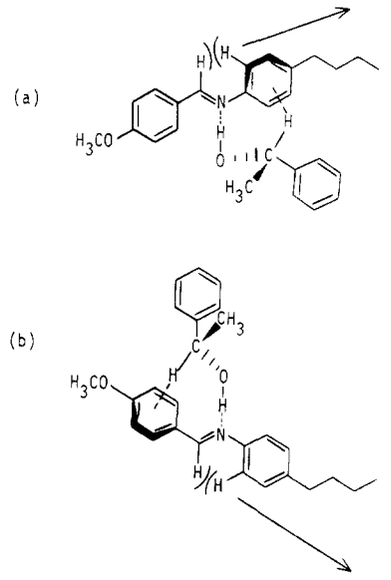


Figure 2. Proposed mode of interaction of (*S*)-**11** with MBBA. Primary interaction of **11** with MBBA occurs between the hydroxyl proton and the lone pair of electrons on nitrogen. Carbinyl hydrogen bonding of **11** to one of the aryl rings of MBBA completes the interaction. The two remaining substituents position themselves more or less perpendicular to the long axis of MBBA (arrow). The least repulsive of these protruding substituents influences the alignment of adjacent MBBA molecules. In the illustration, the methyl group allows adjacent MBBA molecules to skew and form a segment of a right-handed helix.

It will be noted that trifluoromethyl substituents appear more "repulsive" than phenyl (but not naphthyl, etc.) by this criterion. Trichloromethyl is more repulsive than phenyl presumably as a consequence of both its size and electronic nature. No cognizance is taken presently of relative "intercalation" tendencies of the various groups. Data for **1cc**, taken from Gottarelli's work, cannot be rationalized by either the original or the modified model.

Despite the apparent overall success of this simple model, the actual situation is apt to prove rather more complex. For example, in the foregoing discussion, the MBBA chromophore is presumed to be planar and achiral. In reality, a certain amount of twisting of this chromophore into chiral conformations can be tolerated without serious disruption of the orbital overlap of the conjugated system. Ordinarily, the *average* conformation will be planar and achiral. However, interaction with a chiral dopant might induce an average conformational chirality (Figure 2), thereby giving rise to the observed helical macrostructure. We consider this hypothesis to be quite plausible.

In our view, the value of the simple model for secondary alcohol promulgated LCICD is that it illustrates and rationalizes the systematic behavior of such systems and not that it is necessarily an accurate description of the origins of this behavior. The present model does suggest future experimental tests of its validity, however.

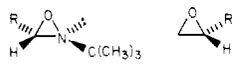
Oxaziridines. We recently had need to assign absolute configurations to partially resolved oxaziridines **2e** and **2f**.[9] The absolute configurations of **2a–d** were known, as were the relative configurations of **2e** and **2f**. Determination of the configurations of **2e** and **2f** relative to those of **2a–d** would hence constitute determination of absolute configurations for **2e–f**. In this context, the LCICD's of dilute MBBA solutions of (−)-enriched oxaziridines **2a–f** were determined. From the uniformly negative LCICD's observed (Table II), it was empirically inferred that (−)-**2e–f**

(7) W. H. Pirkle, S. D. Beare, and R. L. Muntz, *J. Am. Chem. Soc.*, **91**, 4575 (1969).

(8) (a) W. H. Pirkle and J. R. Hauske, *J. Org. Chem.*, **42**, 1839 (1977), and references cited therein; (b) J. D. Morrison and H. S. Mosher, "Asymmetric Organic Reactions", Prentice-Hall, Englewood Cliffs, N.J., 1971, p 191.

(9) W. H. Pirkle and P. L. Rinaldi, *J. Org. Chem.*, **43**, 4475 (1978).

Table II. LCICD of Oxaziridines and Epoxides in MBBA



2 3

compd	R	induced CD,[a] 2% in MBBA
2a	CH_3	-
2b	$CH(CH_3)_2$	-
2c		-
2d	$PhCH_2$	-
2e	4-BrPh	-
2f	1-naphthyl	-
3a	Ph	+
3b	*n*-octyl	-
3c	C_2H_5	-
3d	CH_3	-

[a] From MBBA chromophore at 410-430 nm.

were of the same absolute configuration (i.e., 2*S*,3*S*) as (-)-**2a-d**. This conclusion was supported by NMR-chiral solvating agent studies.[9]

Epoxides. The LCICD data for several (*R*)-(+)-epoxides[10] appears in Table II. Although too little data are at hand to warrant discussion, it is apparent that **3a**, styrene oxide, behaves differently than the alkyl-substituted epoxides **3b-d**.

Conclusion. Evidence is offered to support the contention that LCICD is systematic and that it may be possible to use the technique as a means of reliably assigning absolute configurations. In any event, the sensitivity of the technique makes it valuable even now as a chirality amplifier. For example, various chiral insect pheromones have been isolated in amounts too small to allow chiroptic measurements. In such instances, the absolute configuration of the natural pheromone might be determined through comparison of its LCICD with that of suitable configurationally established model compounds. These model compounds might be synthetic samples of the pheromone enantiomers.

Experimental Section

Circular dichroism spectra were obtained by using a Jasco J-40 spectrometer, and optical rotations were obtained by using a Zeiss visual polarimeter and a 1-dm tube.

Chiral Alcohols 1a-q. These alcohols were synthesized and resolved by procedures previously described.[11-14] The characterization and absolute configuration determination for the following alcohols have previously been reported: **1a** and **1b**;[14] **1o** and **1p**;[11] **1c-j**;[13] **1g** and **1k**.[14] **1m** and **1n** were provided by Mr. K. Simmons, University of Illinois. Their configurations were determined as previously described.[8a]

Oxaziridines 2a-f. Oxaziridines were synthesized enantiomerically enriched (15-60%) by monoperoxycamphoric acid oxidation of the corresponding imines, as previously described.[9]

Epoxides 3a-d. These epoxides were prepared and resolved as previously described.[10]

Examination of LCICD in MBBA. Solutions of chiral substrate were made by adding the substrate (~2% by weight) to MBBA and heating the mixture above the liquid-crystal-liquid transition temperature (~40 °C). One drop of this solution was placed between two glass slides with 24-μm Mylar spacers. The mounted plates were placed in the CD instrument and allowed to cool. As the cholesteric mesophase formed, optical activity could be detected in the region of 390-410 nm. The CD spectrum was scanned from 700 to 200 nm. Although no pitch bands were ever observed, an intense CD band associated with the conjugated imino group of MBBA was always apparent at 390-410 nm. The above procedure was repeated a total of 3 to 5 times to eliminate the possibility that the observed CD band was a result of mechanical twisting of the plates. The importance of this precaution cannot be overstressed. We originally misassigned the LCICD sign for *S*-(+)-**1a** (**1a** gives a weaker than usual LCICD) and noted the error only after Professor Gottarelli informed us of his differing assignment for **1a**. Runs without chiral dopant afforded only weak random CD activity of mechanical origin.

Acknowledgment. This work was partially supported by grants from the National Science Foundation and the National Institutes for Health.

Registry No. (*S*)-(+)-**1a**, 340-06-7; (*S*)-(+)-**1b**, 33758-06-4; (*S*)-(+)-**1c**, 60646-30-2; (*S*)-(+)-**1d**, 63017-54-9; (*S*)-(+)-**1e**, 59153-46-7; (*S*)-(+)-**1f**, 73048-43-8; (*S*)-(+)-**1g**, 73048-44-9; (*S*)-(+)-**1h**, 73048-45-0; (*R*)-(-)-**1i**, 73048-46-1; (*S*)-(+)-**1j**, 59153-45-6; (*S*)-(+)-**1k**, 53432-38-5; (*S*)-(-)-**1m**, 73048-47-2; (*S*)-(-)-**1n**, 73048-48-3; (*S*)-(+)-**1o**, 73048-49-4; (*R*)-(-)-**1p**, 73048-50-7; (*R*)-(-)-**1q**, 73048-51-8; **2a**, 62107-41-9; **2b**, 67504-37-4; **2c**, 67425-89-2; **2d**, 67425-90-5; **2e**, 62058-74-6; **2f**, 67425-91-6; **3a**, 20780-53-4; **3b**, 67210-36-0; **3c**, 3760-95-0; **3d**, 15448-47-2.

(10) W. H. Pirkle and P. L. Rinaldi, *J. Org. Chem.*, **43**, 3803 (1978).
(11) T. G. Burlingame, Ph.D. Thesis, University of Illinois, Urbana, IL, 1970.
(12) W. H. Pirkle, D. L. Sikkenga, and M. S. Pavlin, *J. Org. Chem.*, **42**, 384 (1977).
(13) M. S. Pavlin, Ph.D. Thesis, University of Illinois, Urbana, IL, 1977.
(14) W. H. Pirkle and M. S. Hoekstra, *J. Magn. Reson.*, **18**, 396 (1975).

Memory of Synthesized Vinyl Polymers for Their Origins[1]

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We report herein studies on the memory of a highly cross-linked styrene-divinylbenzene copolymer for its origins. Specific experiments are described which indicate that vinyl polymers synthesized from diastereoisomers of truxinic and truxillic acid are able to recognize the stereoisomer appended during their synthesis, in subsequent chemical transformations. Specific characteristics of polymer cross-link density and other factors as related to the specificity of recognition are outlined.

Nature's "trial and error" chemistry over an enormous length of time, conducted in tremendous numbers, has led to ingenious and refined systems. Efficient catalysts allow reactions to proceed under ordinary conditions, whereas

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