protons were accounted for from the ^{13}C data. The remaining five protons were concluded to be exchangeable protons, but only four could be detected in the ^{1}H NMR spectrum in benzene-d_6.

Ten partial structures, units A–J (Figure 1), were generated mainly from 2D ^{1}H NMR, viz. COSY,[9] Relay (RCT1),[10] Double Relay (RCT2),[10] TOCSY,[11] phase-sensitive NOESY,[12] and ROESY[13a,b] experiments, and ^{13}C chemical shift data (Table I). Sequencing units A–J into a gross structure for **1** was then readily accomplished from proton-detected one-bond and long-range ^{1}H–^{13}C NMR experiments, viz. HMQC[14] and HMBC.[15]

The homonuclear connectivities in the various units were deduced from the 2D data as illustrated in the following two examples. For unit F the H-20 signal showed COSY cross peaks to H-19 and H-21, relay cross peaks to H-22 and H_3-38, and double relay cross peaks to H-23 (weak) and H_3-37; no additional cross peaks were seen in the TOCSY spectrum. In unit B the H_3-43 signal showed a COSY cross peak to H-5, a relay cross peak to H-6b (failure to see cross peaks to H-4a, H-4b, and H-6a indicated small couplings from H-5 to these protons), a double relay cross peak to H-6a and H-7 (weak), and TOCSY cross peaks to H-3a, H-3b, H-4a, H-4b, H-8, and H_3-42. The only proton signal for unit B that was not observed as a cross peak in the TOCSY spectrum was the one for the exchangeable OH proton on C-7.

Four-bond ^{1}H–^{1}H coupling was important in determining the structure of unit G, including the placement of a hydroxyl group on C-24 (COSY shows cross peaks from the OH on C-24 to H-25 and H_3-36), and in connecting a methyl group to C-18 in unit F. Vicinal coupling constants indicated that (1) the geometries of the Δ^{11} and Δ^{20} double bonds were cis and trans, respectively, (2) hydroxyl groups were attached to C-15, C-16, and C-47, and (3) a methyl group was attached to the C-32 methine in an epoxide ring. NOEs established that (1) the geometries of the Δ^{18} and Δ^{25} double bonds were *E* and *Z*, respectively, (2) a second methyl group was attached to the epoxide ring at C-31 and oriented cis to the methyl group on C-32, and (3) the methoxyl group was on C-17.

The chemical shift of H-23 indicated that an ester oxygen was attached to C-23 as shown in unit F. The chemical shift of H-32 further supported its attachment to an epoxide ring as shown in unit H.

After assigning all the protonated carbons by the HMQC experiment, units A–J were easily connected from the HMBC[16] data (see Table II in Supplementary Material). The key two- and three-bond heteronuclear connectivities (^{1}H to ^{13}C) that established the gross structure of **1** were as follows: H_3-44 to C-1; H-3a to C-1, C-2, and C-44; H_3-42 to C-9; H_3-41 to C-9; H-16 to C-15; H-17 to C-15, C-18, C-19, and C-39; H_3-39 to C-17; H-23 to C-1, C-25, and C-36; H-25 to C-23 and C-36; H-27a to C-29; H-29 to C-27, C-28 and C-30; H-32 to C-30 and C-31; H_3-34 to C-30; H-46 to C-45 and C-48; H_3-49 to C-45 and C-47 (indicated by arrows in Figure 1). No direct NMR evidence was obtained to link A to I to J. The only alternative would be to link J to the oxygen on C-7. The chemical shift of H-7 (δ 3.86), however, strongly supported the placement of a hydroxyl on C-7. Unit J, therefore, had to be connected to I, and I in turn to the oxygen on C-2 by process of elimination. Even though vicinal coupling was not observed between H-15 and H-16, an NOE between these two protons as well as two- and three-bond heteronuclear correlations from H-16 and H-17 to C-15 firmly established the attachment of D and E as shown.

Patellazole B appears to be predominantly polyketide-derived, but the thiazole probably originates from an amino acid. Studies on the biosynthesis of patellazole B and the role of the algal symbiont in its production have been initiated.

Acknowledgment. This research was supported by the University of Hawaii Sea Grant College Program under Institutional Grant NA 81AA-D-0070 from NOAA, Office of Sea Grant, U.S. Department of Commerce, and NIH Grants CA12623 (R.E.M.) and GM28624 (V.J.P.). We thank Faith Caplan and Kay Larsen for performing the cytotoxicity tests.

Supplementary Material Available: Table II listing the homonuclear and heteronuclear connectivities for patellazole B and ^{1}H NMR, PSCOSY, RCT1, RCT2, TOCSY, PSNOESY, ROESY, HMQC, and HMBC spectra of patellazole B in benzene-d_6 (0.018 M for all spectra except RCT1 and RCT2) (14 pages). Ordering information is given on any current masthead page.

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[8] Mycothiazole shows carbon signals at 154.9, 111.8, and 179.4 ppm for the thiazole carbons corresponding to C-28, C-29, and C-30 in patellazole B [Crews, P.; Kakou, Y.; Quinoa, E. *J. Am. Chem. Soc.* **1988**, *110*, 4365]. We thank P. Crews for sending us a preprint.

[9] Bax, A.; Freeman, R.; Morris, G. *J. Magn. Reson.* **1981**, *42*, 164.

[10] Bax, A.; Drobny, G. *J. Magn. Reson.* **1985**, *61*, 306. The RCT1 and RCT2 spectra were determined on a 0.01 M solution of patellazole B in benzene-d_6; refocusing delays of 20 ms were used for the two experiments.

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[12] States, D. J.; Haberkorn, R. A.; Ruben, D. J. *J. Magn. Reson.* **1982**, *48*, 286.

[13] (a) Bothner-By, A. A.; Stephens, R. L.; Lee, J.-M.; Warren, C. D.; Jeanloz, R. W. *J. Am. Chem. Soc.* **1984**, *106*, 811. (b) Bax, A.; Davis, D. G. *J. Magn. Reson.* **1985**, *63*, 207.

[14] Bax, A.; Subramanian, S. *J. Magn. Reson.* **1986**, *67*, 565.

[15] Bax, A.; Summers, M. F. *J. Am. Chem. Soc.* **1986**, *108*, 2094.

[16] A mixing time of 70 ms was used to detect 7 Hz coupling. The total acquistion time was 1.5 h.

Unprecedented Stereochemical Control in the Claisen Rearrangement of Allyl Vinyl Ethers Using Organoaluminum Reagents

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Received June 30, 1988

The Claisen rearrangement and its variants (Carroll, the ortho ester, Eschenmoser, and Ireland rearrangements)[1] offer many attractive synthetic advantages in view of the simplicity and versatility as exemplified by the broad applications to the stereo- and regiochemically defined synthesis of a wide variety of natural products.[2] Among these, the basic Claisen rearrangement of vinyl ethers **1** of secondary allylic alcohols affords γ,δ-unsaturated aldehydes in which the *E*-isomer, (*E*)-**2**, invariably predominates with the extent of $E/Z = {\sim}9{:}1$.[3] Apparently, such *E*-selectivity is a general attribute of the Claisen family.[4] The opposite *Z*-

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[1] Reviews: (a) Rhoads, S. J.; Raulins, N. R. *Org. React.* (*N.Y.*) **1975**, *22*, 1. (b) Bennett, G. B. *Synthesis* **1977**, 589. (c) Ziegler, F. E. *Acc. Chem. Res.* **1977**, *10*, 227. (d) Bartlett, P. *Tetrahedron* **1980**, *36*, 2. (e) Hill, R. K. In *Asymmetric Synthesis*; Morrison, J. D., Ed.; Academic Press, New York, 1984; Vol. 3B, p 503. (f) Carruthers, W. *Some Modern Methods of Organic Synthesis*, 3rd ed.; Cambridge University Press: Cambridge, 1986; p 167.

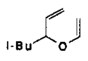
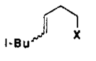
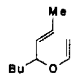
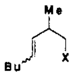
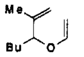
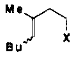
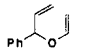
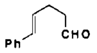
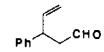
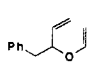
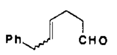
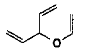
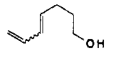
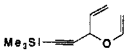
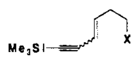
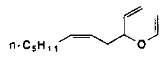
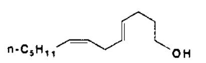
[2] (a) Ireland, R. E.; Thaisrivongs, S.; Wilcox, C. S. *J. Am. Chem. Soc.* **1980**, *102*, 1155. (b) Danishefsky, S.; Tsuzuki, K. *Ibid.* **1980**, *102*, 6891. (c) Ireland, R. E.; Wuts, P. G. M.; Ernst, B. *Ibid.* **1981**, *103*, 3205. (d) Martinez, G. R.; Grieco, P. A.; Williams, E.; Kanai, K.; Srinivasan, C. V. *Ibid.* **1982**, *104*, 1436. (e) Bartlett, P. A.; Barstow, J. F. *J. Org. Chem.* **1982**, *47*, 3933. (f) Bartlett, P. A.; Tanzella, D. J.; Barstow, J. F. *Ibid.* **1982**, *47*, 3941. (g) Ireland, R. E.; Daub, J. P.; Mandel, G. S.; Mandel, N. S. *Ibid.* **1983**, *48*, 1312. (h) Ireland, R. E.; Courtney, L.; Fitzsimmons, B. J. *Ibid.* **1983**, *48*, 5186. (i) Kallmerten, J.; Gould, T. J. *Ibid.* **1986**, *51*, 1155. (j) Kallmerten, J.; Balestra, M. *Ibid.* **1986**, *51*, 2857. (k) Barrish, J. C.; Lee, H. L.; Baggiolini, E. G.; Uskokovic, M. R. *Ibid.* **1987**, *52*, 1375.

[3] (a) Brannock, K. C. *J. Am. Chem. Soc.* **1959**, *81*, 3379. (b) Burgstahler, A. W. *Ibid.* **1960**, *82*, 4681.

[4] In the Eschenmoser, Ireland, and the ortho ester rearrangements, the exclusive *E*-selectivity is observed: (a) Faulkner, D. J.; Petersen, M. R. *J. Am. Chem. Soc.* **1969**, *95*, 553. (b) Faulkner, D. J.; Peterson, M. R. *Tetrahedron Lett.* **1969**, 3243. (c) Katzenellenbogen, J. A.; Christy, K. J. *J. Org. Chem.* **1974**, *39*, 3315. (d) Ireland, R. E.; Mueller, R. H.; Willard, A. K. *Ibid.* **1976**, *41*, 986. See, also: (e) Kimel, W.; Cope, A. C. *J. Am. Chem. Soc.* **1943**, *65*, 1992. (f) Hill, R. K.; Synerholm, M. E. *J. Org. Chem.* **1968**, *33*, 925. (g) Wakabayashi, N.; Waters, R. M.; Church, J. P. *Tetrahedron Lett.* **1969**, 3253. (h) Johnson, W. S.; Werthemann, L.; Bartlett, W. R.; Brocksom, T. J.; Li, T.; Faulkner, D. J.; Petersen, M. R. *J. Am. Chem. Soc.* **1970**, *92*, 741.

0002-7863/88/1510-7922$01.50/0

able I. Organoaluminum-Promoted Claisen Rearrangement[a]

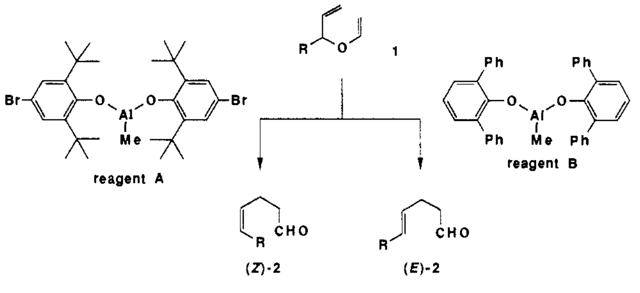
entry	substrate	reagent[b]	product[c]	yield[d] (%)	ratio (E/Z)[e,f]
		→			
1		A	X = CHO	64	7:93
2		B	X = CH_2OH	85	97:3 (92:8)
3		C	X = CH_2OH	55	87:13
		→			
4		A	X = CHO	72	16:84
5		A[g]	X = CHO	70	12:88
6		B	X = CH_2OH	94	99:1 (92:8)
		→			
7		A	X = CHO	58	61:39
8		B	X = CH_2OH	94	95:5 (83:17)
		→		+	
9		A	31%		49%
10		B	45%		13%
11		A		12	40:60
12		B		95	92:8
13		B[h]		78	99:1
14		A		97	24:76
15		B		91	90:10
		→			
16		A	X = CHO	94	3:97
17		B	X = CH_2OH	82	69:31
18		B	 3	88	95:5
19		B[h]		79	98:2 (93:7)

[a] The rearrangement was effected by treatment of a substrate with the modified organoaluminum reagent A or C (2 equiv) at −78 °C for 15–30 min or with the reagent B (2 equiv) at −20 °C for 15 min. For experimental details, see Supplementary Material. [b] Reagent C: diisobutylaluminum -bromo-2,6-di-*tert*-butylphenoxide. For structures of the reagents A and B, see text. [c] When the aluminum reagent B was utilized, olefinic aldehydes were generally reduced to the corresponding alcohols with $NaBH_4/MeOH$ in view of the easy product separation from 2,6-diphenylphenol. Isolated yield by column chromatography. [e] In general, the E/Z ratios were determined by GLC after converting to the alcohol ($NaBH_4/MeOH$) and then to the trimethylsilyl ethers ($Me_3SiCl-NEt_3/CH_2Cl_2$). [f] The E/Z ratios in parentheses refer to those in the thermal Claisen rearrangement 250 °C). [g] At −95 °C. [h] At −78 °C.

selectivity, however, has never been achievable so far for lack of suitable methodologies. Here we wish to disclose the first stereocontrolled Claisen rearrangement of allyl vinyl ethers of type **1** with certain bulky organoaluminum reagents. This method serves as a stereoselective route to γ,δ-unsaturated aldehydes with the E- or Z-configuration by appropriately modifying the organoaluminum ligands (Scheme I).[5,6]

When allyl vinyl ether **1** (R = *i*-Bu) was treated with methylaluminum bis(4-bromo-2,6-di-*tert*-butylphenoxide) (reagent A) in CH_2Cl_2 at −78 °C, the rearrangement proceeded smoothly to furnish 7-methyl-4-octenal (**2**) (R = *i*-Bu) in 64% yield. The E/Z ratio of **2** was determined to be 7:93 by capillary GLC after conversion of the aldehyde to the corresponding alcohol and then to the trimethylsilyl ether. In marked contrast, however, the rearrangement of **1** (R = *i*-Bu) in toluene under the influence of methylaluminum bis(2,6-diphenylphenoxide) (reagent B) at −20 °C gave rise to the E-isomer, (E)-**2** (R = *i*-Bu) almost exclusively (E/Z = 97:3) in 85% yield. The generality of the present stereocontrolled Claisen rearrangement is indicated in Table I. Several characteristic features have been noted. (1) In general, the reagent A can be utilized for obtaining the Z-isomer, while the E-isomer was produced with the reagent B. (2) Compared to the conventional thermal rearrangement that requires high temperature, the organoaluminum-promoted Claisen rearrangement transpires under very mild experimental conditions, particularly at strikingly low temperatures with eminent E- and Z-selectivities. The observed E-selectivity is mostly superior to that in the thermal rearrangement (Table I). (3) Both E- and Z-selectivities appear to increase by lowering the reaction temperature. Reaction of the substrate **1** (R = *i*-Bu) with the reagent A or B at 0 °C resulted in the E/Z ratio of 17:83 or 83:17, respectively. (4) The *p*-bromo substituent in the reagent A is indispensable for rate acceleration of the rearrangement. At-

Scheme I



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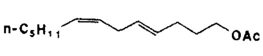
[5] The Lewis acid catalyzed Claisen rearrangements of simple allyl vinyl ethers have appeared recently. (a) Nonstereoselective rearrangement of vinyl ethers of secondary alcohols with organoaluminum reagents: Takai, K.; Mori, I.; Oshima, K.; Nozaki, H. *Tetrahedron Lett.* **1981**, *22*, 3985. (b) Pd-catalyzed rearrangement of vinyl ethers of primary alcohols: van der Baan, J. L.; Bickelhaupt, F. *Ibid.* **1986**, *27*, 6267.

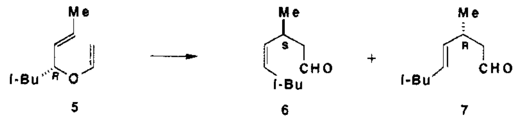
[6] The Z-selective [2,3]-Wittig rearrangement of zirconium enolates of alkenyloxyacetic acid esters: Uchikawa, M.; Katsuki, T.; Yamaguchi, M. *Tetrahedron Lett.* **1986**, *27*, 4581.

tempted use of methylaluminum bis(2,6-di-*tert*-butyl-4-methylphenoxide) (MAD) significantly retarded the rate of the reaction. (5) The rearrangement using the reagent A in toluene somewhat lowered the *Z*-selectivity. A similar propensity for *E*-selectivity was observed with the reagent B in CH_2Cl_2 in lieu of toluene. (6) In case of the substrate **1** (R = Ph), [1,3]-sigmatropic rearrangement took place in competition to the normal [3,3]-Claisen rearrangement (entries 9 and 10). (7) 2-Methyl-1-hepten-3-yl vinyl ether gave the *E*-isomer as a major product even with the reagent A (entry 7). (8) The conjugated *Z*-enyne structural units, which are often present in biologically active natural products,[7] can be readily available by this approach (entry 16). (9) Entries 18 and 19 illustrate the synthetic utility of the present reaction in natural product synthesis. The Claisen product **3**[8] is readily transformed by simple acetylation into (4*E*,7*Z*)-4,7-tridecadienyl acetate (**4**), a component of the sex pheromone of potato tuberworm moth (*Phthorimaea operculella*).[9]

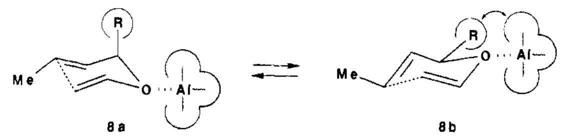


4

Examination of the stereochemical aspect in the rearrangement of the optically active substrate **5** is of special interest in order to elucidate the transition state in the organoaluminum-promoted Claisen rearrangement. Thus, individual treatment of **5** with the reagents A and B under the standard conditions as described above gave the (*S*)-(*Z*)-aldehyde **6** and the (*R*)-(*E*)-aldehyde **7**, respectively as major products with moderate transfer of the chirality of **5** (74–78% chiral transmission).[10,11] Consequently, the observed selectivities are best accounted for by the two possible chairlike transition-state conformations **8a** and **8b** coordinated to the Lewis acidic aluminum reagent.[12] The conformation **8a**, with the R substituent axial, is thought to be highly unfavorable in the Claisen rearrangement and its variants.[1] However, the less likely **8a**, when complexed with the exceptionally bulky organoaluminum reagent, would be favored over **8b** in view of the severe 1,2-steric interaction between R and the aluminum reagent in **8b**, leading to the preferential formation of (*Z*)-alkene. In fact, when the bulkiness of the aluminum reagent is decreased from the reagent A to dimethylaluminum 4-bromo-2,6-di-*tert*-butylphenoxide, the *E*/*Z* selectivity in the rearrangement of the substrate **1** (R = *i*-Bu) is changed dramatically from 7:93 to 71:29, suggesting that the population of the transition state shifts from **8a** to **8b** by decreasing the steric size of aluminum ligands. The origin of the exceedingly high *E*-selectivity with the reagent B, which remains unclear at present, seems to imply the importance of the electronic factor of the 2,6-diphenylphenoxy ligand as well as its steric factor.



reagent A : 74% (84 : 16)
reagent B : 90% (2 : 98)



Supplementary Material Available: Details for determination of the absolute configurations and the optical purities of **6** and **7** and experimental procedures for the *Z*- and *E*-selective Claisen rearrangements (2 pages). Ordering information is given on any current masthead page.

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[7] For example, see: (a) Witkop, B. *Experientia* **1971**, *27*, 1121. (b) Tokuyama, T.; Uenoyama, K.; Brown, G.; Daly, J. W.; Witkop, B. *Helv. Chim. Acta* **1974**, *57*, 2597. (c) Guerreno, A.; Camps, F.; Coll, J.; Riba, M.; Einhorn, J.; Descoins, Ch.; Lallemand, J. Y. *Tetrahedron Lett.* **1981**, *22*, 2013.

[8] We appreciate Dr. S. Tsuboi of Okayama University for providing valuable information on the preparation of the starting allylic vinyl ether.

[9] Previous synthesis of **4**: (a) Roelofs, W. L.; Kochansky, J. P.; Carde, R. T.; Henrick, C. A.; Labovitz, J. N.; Corbin, V. L. *Life Sci.* **1975**, *17*, 699. (b) Voerman, S.; Rothchild, J. *J. Chem. Ecol.* **1978**, *4*, 531. (c) Alexakis, A.; Cahiez, G.; Normant, J. F. *Tetrahedron Lett.* **1978**, 2027. (d) Fujisawa, T.; Sato, T.; Kawashima, M.; Naruse, K.; Tamai, K. *Ibid.* **1982**, *23*, 3583. (e) Nishiyama, H.; Sakuta, K.; Itoh, K. *Ibid.* **1984**, *25*, 223. (f) Vig, O. P.; Sharma, M. L.; Kumari, S.; Rani, V. *Indian J. Chem., Sect. B* **1985**, *24B*, 675. (g) Yadav, J. S.; Reddy, P. S. *Synth. Commun.* **1986**, *16*, 1119. (h) Yadav, J. S.; Kulkarni, A. D.; Reddy, P. S. *Indian J. Chem., Sect. B* **1986**, *25B*, 1220. (i) Chattopadhyay, A.; Mamdapur, V. R.; Chadha, M. S. *Ibid.* **1987**, *26B*, 187.

[10] The ratios of **6** and **7** were determined by capillary GLC after transformation of the Claisen products to the corresponding alcohols and then to the trimethylsilyl ethers.

[11] The absolute configurations of the Claisen products were determined by correlation to optically active citronellal. See Supplementary Material.

[12] The possibility of the boatlike transition-state conformation with the R substituent equatorial, which leads to (*Z*)-alkene, may not be excluded. However, according to the ab initio quantum mechanical calculations the intervention of the boatlike transition structure seems to be unlikely because of the high energy compared to the chairlike transition structure. See: Vance, R. L.; Rondan, N. G.; Houk, K. N.; Jensen, F.; Borden, W. T.; Komornicki, A.; Wimmer, E. *J. Am. Chem. Soc.* **1988**, *110*, 2314.

Evidence for Dipolar Cross-Correlation from Triple-Quantum-Filtered Two-Dimensional Exchange NMR Spectroscopy

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Received June 6, 1988

Two-dimensional nuclear Overhauser effect spectroscopy (NOESY) is now extensively used for structural studies in a large variety of systems.[1-3] The interpretation of NOESY spectra is usually based on Solomon's equations,[4] although these are merely intended to describe the exchange of the two longitudinal Zeeman terms $\langle I_{kz}\rangle$ and $\langle I_{lz}\rangle$ due to cross-relaxation in systems containing only two spins. In a system with three nuclei, however, the relaxation behavior[5-7] can only be fully described by a set of four differential equations, which describe the coupling of the three terms $\langle I_{kz}\rangle$, $\langle I_{lz}\rangle$, $\langle I_{mz}\rangle$, and of longitudinal three-spin order $\langle 4I_{kz}I_{lz}I_{mz}\rangle$. While $\langle I_{kz}\rangle$ and $\langle I_{lz}\rangle$ are coupled through the usual cross-relaxation rate σ_{kl} (which depends only on auto-correlation spectral density functions), $\langle I_{kz}\rangle$ and $\langle 4I_{kz}I_{lz}I_{mz}\rangle$ are coupled by a rate δ_{klkm} that depends on cross-correlation of the dipolar *kl* and *km* interactions. If the molecular motion is isotropic, the rate of the conversion from $\langle I_{kz}\rangle$ into $\langle 4I_{kz}I_{lz}I_{mz}\rangle$ is given by[5-8]

$$\delta_{klkm} = c\langle r_{kl}\rangle^{-3}\langle r_{km}\rangle^{-3}P_2(\cos\theta_{klkm})\tau_c/(1 + \omega_k^2\tau_c^2) \qquad (1)$$

where $c = (^3/_5)\,(\mu_0/4\pi)^2\gamma^4\hbar^2$ and $P_2(\cos\theta_{klkm}) = {}^1/_2(3\cos^2\theta_{klkm} - 1)$, θ_{klkm} being the angle subtended by the internuclear vectors $\mathbf{r}_{kl}$ and $\mathbf{r}_{km}$. Measuring the initial build-up rate of $\langle 4I_{kz}I_{lz}I_{mz}\rangle$ allows one to determine δ_{klkm}.

The purpose of this communication is to present experimental evidence that longitudinal three-spin order can be separated from the Zeeman terms and that two-dimensional spectroscopy provides a suitable tool for studying cross-correlation.

Three-spin order (or, analogously, octupolar order) has been observed in a selective manner in $^{13}CH_2$ systems by Brondeau et al.,[9] in ^7Li ($S = {}^3/_2$) by Jaccard et al.,[10] in methyl groups by

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