radical and again suggests that hydrogen abstraction by bromine atom is not the major chain-propagating step.

Conclusion

Photolytic bromination at benzylic positions by diethyl bromomalonate is a slow process probably indicative of short radical chain length. Despite this, it appears that the major chain-propagating species is the diethyl malonyl radical. This species shows sensitivity toward electronic changes which are typical of electrophilic radicals. Its size, however, causes it to be very selective with regard to reaction at hindered benzylic sites. Unfortunately, the present data does not allow a more detailed analysis of the differences between the two radicals to be made. The relative positions of the benzylic hydrogen atom abstraction by bromine atom and diethyl malonyl radical can vary on the reaction coordinate. An additional complication is due to the known reversibility of the former process.[11]

Experimental Section

Compounds. The majority of compounds used in this study were commercially available. The preparation of specific substituted toluenes,[10] substituted cumenes,[15] and bromo esters[7] have been discussed in earlier papers from this laboratory. All compounds were purified by standard procedures before use.[16]

Kinetics. All kinetic studies were run on pairs of compounds. The reactions were run in a thermostated oil bath. The reaction times were varied to ensure from 4% to 75% reaction for each substrate. The reaction mixtures were divided among several ampoules which were then sealed under a reduced pressure of nitrogen after three freeze–thaw cycles. In each case, one of the sealed ampoules was reserved for analysis of starting material. Reaction mixtures contained a pair of compounds, an internal standard (either 1,2-dichlorobenzene or chlorobenzene), DEBM, and benzene in an approximate molar ratio of 1:1:1:1:5. The ampoules were placed in the bath, just below the surface of the oil, and irradiated with a GE 275-W sun lamp. Reaction times varied from 15 to 141 h.

Analysis. All analyses were carried out using one of the following gas chromatographs: a HP 5730A equipped with a flame ionization detector and a 10-ft × $^{1}/_{4}$-in. 12% Carbowax 20M column, a HP 5710A equipped with a thermal conductivity detector and a 6-ft ×$^{1}/_{8}$-in. 10% SP2100 column, or a Varian 3400 equipped with a flame ionization detector, autosampler, and a DB-17 capillary column.

Acknowledgment. One of us (S.V.T.) wishes to thank the administrators of the N. L. Tartar Fellowship for providing financial support. We are indebted to Dr. P. K. Freeman for the use of the capillary gas chromatograph.

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A Regioselective Decarbonylation

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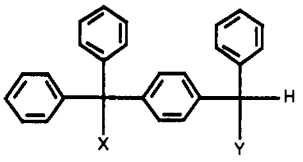
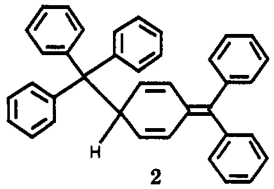
Carboxylic acid dianion oxidative coupling provides convenient synthetic methodology for the construction of certain natural products.[1] In a mechanistic study on oxidative coupling of systems related to phenylacetic acid dianion, Fox reported[2] isolation of an unusual reaction product 1 from oxidative coupling of the hindered diphenylacetic acid dianion. Formation of diacid 1 is reminiscent of the major product 2 produced[3] during attempted preparation[4] of hexaphenylethane.

Compound 1 serves as an easily accessible substrate with which to compare the chemical reactivity of two carboxylic acid groups that are located within the same molecule but which have obviously different steric and electronic environments. To probe this issue, we sought to ascertain whether or not the two carboxylic acid groups in 1 would exhibit distinguishable behavior in the presence of concentrated sulfuric acid. Solvation and protonation effects of sulfuric acid as a reaction medium have been the subject of a monograph.[5]

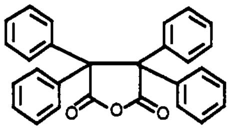
Crude samples of 1 containing siginificant amounts (5–10%) of both polymer and diacid 4 result from treatment of diphenylacetic acid dianion with molecular iodine.[2] Similar solubility and polarity properties of 1 and 4 as well as the presence of polymeric byproducts necessitated a simple, if tedious, purification protocol. Trituration of crude 1 with a mixture of ether and ligroin followed by addition of excess diazomethane to the residue[2] provided a reaction mixture that is predominantly diester 5. Further purification of 5 by chromatography, saponification under forcing conditions, and a final recrystallization then affords analytically pure diacid 1. Since diacid 4 should readily undergo loss of water,[6] consistent obtention of 3 as a newly formed nonpolar dehydration product (chromatographed yield 8%) upon admixture of crude 1 and warm concentrated H_2SO_4 confirms the presence of 4 in crude 1.





2

	X	Y
1	COOH	COOH
5	COOCH$_3$	COOCH$_3$
6	OH	COOH
7	OH	COOCH$_3$
8	Cl	COCl
9	OCH$_3$	COOCH$_3$
10	OEt	COOEt
11	OH	COOEt



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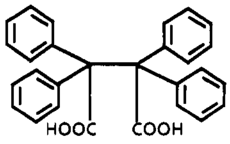

4

Using carefully purified 1, optimal conditions for the

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[4] Gomberg, M. *Chem. Ber.* 1900, *33*, 3150.

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[6] For prior indications that hindered succinic acids are especially prone to undergo dehydration, see: Belletire, J. L.; Conroy, G. *Synth. Commun.* 1988, *18*, 403. In her work,[2] Fox reports the production of 1 and 3 (as a trace) from the electrolysis of the diphenylacetic acid dianion.

0022-3263/92/1957-2970$03.00/0

Table I

		relative abundance after specified times *t*					
peaks	*m/z*	control	7 min	13 min	20 min	25 min	30 min
N_2 or CO	28	300	873	1000	1000	1000	1000
Ar	40	1000	1000	758	369	327	229
ratio *m/z* 28/*m/z* 40		0.3	0.9	1.3	2.7	3.1	4.4

decarbonylation were developed. Relatively clean reaction mixtures were obtained after treatment with concentrated sulfuric acid for 6–7 min at 45 °C in contrast to the more complex reaction mixtures observed upon treatment with concentrated sulfuric acid for longer periods at room temperature or below. Upon addition of the powdered diacid 1 to sulfuric acid that had been prewarmed to 45 °C, there occurs an initial vigorous effervescence that gradually diminishes in intensity as the reaction mixture darkens to brownish-red. After workup, preparative TLC provides 6 as a glass that slowly crystallizes (52% yield).

By carrying out the sulfuric acid treatment using a closed system under an argon atmosphere, CO was identified by GC/MS as the gas liberated after addition of 1 to preheated concentrated sulfuric acid. Regioselective formation of 6, with sequential liberation of water and CO after initial protonation, is in accord with the accepted mechanism[5] for such decarbonylations wherein an acylium ion intermediate is produced. This species then undergoes loss of carbon monoxide to afford a triarylmethyl carbocation that, in turn, survives until a nucleophile (e.g. water) is added. Other acids, such as 12 M aqueous HCl, polyphosphoric acid, and neat HCOOH were explored as reaction media in place of the concentrated sulfuric acid, but their use led only to recovered starting diacid 1. Stereochemical equilibration and reversible addition or loss of CO to carbocation intermediates in sulfuric acid/formic acid mixtures are key mechanistic features of the closely related Koch–Haaf reaction.[7-10]

Chemical evidence by formation of simple derivatives (without attempts at optimization) was also obtained to support the identity of 6. Treatment of 6 with excess diazomethane gave the expected hydroxy ester 7. Conversion of hydroxy acid 6 into the corresponding chloro acid chloride 8 was accomplished by prolonged heating of 6 in benzene with an excess of oxalyl chloride. When acid chloride 8 was mixed with a THF solution of excess methanol and excess pyridine, the corresponding ether–ester 9 could be isolated as a colorless oil in good yield (69%) by preparative TLC. Treatment of acid chloride 8 with a THF solution of excess ethanol and excess pyridine yields the ether–ester 10 in moderate yield (47%) after preparative TLC. Addition of acid chloride 8 to ethanol in the absence of pyridine followed by an aqueous workup affords, after preparative TLC, a mixture of the corresponding ether–ester 10 (28%) and the hydroxy ester 11 (48%) (chromatographed yields based on acid chloride starting material). All of these results are consistent with identification of 6 as a triarylmethanol derivative.[11]

Experimental Section

All solvents and reagents were commercially available reagent grade. THF was freshly distilled under nitrogen from benzophenone sodium ketyl. NMR proton spectra were run on a Bruker Model AC-250 250-MHz spectrometer which was then routinely switched over for the collection of completely decoupled carbon spectra. IR spectra were obtained on a Perkin-Elmer Model 1600 FT instrument. Low-resolution GC/MS scans were obtained on a Perkin-Elmer Model 5995 instrument while high-resolution mass spectra were obtained on a Kratos Model MS-801 DS55 spectrometer. TLC analyses were done on analytical plates having a fluorescent indicator using development by 10–20% EtOAc/80–90% ligroin with UV or iodine visualization in addition to a dip in bromcresol green to indicate spots corresponding to carboxylic acid. Preparative TLC was done using 20- × 20- × 0.2-cm plates having fluorescent indicator. Multiple development with EtOAc/ligroin mixtures was then followed by mechanical removal of the desired band and elution with EtOAc.

α,α,α'-Triphenyl-1,4-benzenediacetic Acid (1). To diphenylacetic acid (6.37 g, 30 mmol) dissolved in 75 mL of dry THF cooled to −78 °C was added dropwise *n*-BuLi solution (1.5 M) in hexane (41.9 mL, 62.8 mmol) over 1 h. The resulting yellow solution was stirred and gradually warmed to rt over 3 h. The reaction mixture was then recooled to −78 °C, and a solution of molecular iodine (3.801 g, 15.0 mmol) in 30 mL of dry THF was added rapidly. The reaction mixture was then stirred for 18 h with gradual warming to rt. After removal of volatiles, the residue was acidified with 70 mL of 1 M HCl. The aqueous suspension was extracted with three 60-mL portions of EtOAc. The organic layers were combined, washed with water (50 mL), saturated sodium bisulfite (50 mL), and saturated brine (50 mL), and dried over $MgSO_4$, and volatiles were removed to afford a white powder. After trituration with hot diethyl ether and ligroin, the residue (mp (with gradual decomposition) 214–238 °C) was semipure diacid (estimated by its further behavior vide infra and by simple TLC analysis to consist of 80% or more of the desired diacid 1) (3.80 g, 60%). By TLC, anhydride 3 was absent.

An ether solution of diazomethane (prepared from *N*-(nitrosomethyl)urea (2.50 g, 24.2 mmol), ether (50 mL), and KOH (3.0 g, 53.5 mmol)) and water (47 mL), after drying over solid KOH, was poured onto semipure diacid 1 (0.50 g, 1.18 mmol) in methylene chloride (30 mL). After 15 h, the volatiles were removed to provide a faint yellow oil with some suspended white crystals. The crude diester was purified by prep TLC (development (twice) with 70:30 ligroin/EtOAc: the desired band is the second least polar) with exhaustive extraction by EtOAc to give 0.330 g (62%). The diester could be further purified by recrystallization (*i*-PrOH/ligroin) to afford analytically pure 5 (mp 136–137 °C (lit.[2] mp 140–141 °C)): IR ($CHCl_3$) 1732 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.32–7.12 (m, 19 H), 5.00 (s, 1 H), 3.74 (s, 3 H), 3.69 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 174.0, 172.8, 142.7, 141.7, 138.3, 137.2, 130.5, 130.1, 128.6, 127.7, 127.3, 126.9, 67.1, 56.5, 52.6, 52.2; MS (EI, 70 eV) *m/z* 59, 91, 119, 157, 181, 241, 332, 391 (100); HRMS ($M^+ - CO_2CH_3$) calcd 391.1698, found 391.1706. Anal. Calcd for $C_{30}H_{26}O_4$: C, 79.98; H, 5.82. Found: C, 79.84. H, 5.88.

The purified diester (1.05 g, 2.33 mmol) was dissolved in 25 mL of THF. To this was added 2.5 M aqueous NaOH solution (30 mL). The resulting mixture was heated at reflux for 48 h. After cooling of the mixture to room temperature, the THF was removed at reduced pressure, and the residue was acidified with concentrated HCl and extracted with EtOAc (3 × 20-mL portions). The combined organic layers were washed with water (20 mL) and with saturated brine (20 mL), dried, and filtered, and the volatiles were removed. The residue was recrystallized from ether/ligroin to give pure diacid 1 (0.703 g, 71%) (mp 209–211 °C): IR ($CHCl_3$) 3400–2500 (br), 1707, 1600, 1495 cm^{-1}; ^1H NMR ($CDCl_3$/d_6-DMSO) δ 7.37–7.10 (m, 19 H), 4.97 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 175.5, 174.6, 143.8, 142.1, 139.6, 138.0, 130.5 130.2, 128.7, 128.5, 127.9, 127.6, 127.1, 126.8, 67.5, 57.1; MS (EI, 70 eV) *m/z* 77, 91, 105, 167 (100), 194, 241, 348, 377; HRMS (M^+ − COOH)

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calcd 377.1541, found 377.1540.

Tetraphenylsuccinic Anhydride (3). Semipure diacid that had been isolated from dianion oxidative coupling by simple trituration (1.00 g, 2.37 mmol) was added to prewarmed (45 °C) concentrated sulfuric acid (8 mL). After 7 min, bubbling had ceased, the reaction mixture was poured over ice and extracted with four 15-mL portions of EtOAc, the combined organic layers were washed twice with 20 mL of water and once with 20 mL of saturated brine, dried, and filtered, and the volatiles were removed. Prep TLC (developing with 30% EtOAc/70% ligroin with elution of the bands by repeated extraction with EtOAc) gave two major components (band "A" (R_f ca. 0.8, 0.080 g, 0.198 mmol, 8.4% based on diacid starting material) that was the largest and middle band of three closely spaced nonpolar bands and band "B" (R_f ca. 0.4, 0.65 g, 1.61 mmol, 68% based on starting material)). Although a trace of colored polymer was present, no diacid remained. Band B was hydroxy acid **6**. Band A was anhydride **3** (mp 207–208 °C). When diacid samples that had been purified via esterification, chromatography, and saponification were treated with concentrated sulfuric acid, the anhydride band was absent. For anhydride **3**: IR ($CHCl_3$) 3011, 1855, 1782, 1496, 1447, 1068, 950 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.21–7.12 (m, 20 H); ^{13}C NMR ($CDCl_3$) δ 171.2, 137.6, 130.6, 128.0, 69.2; MS (EI, 70 eV) *m/z* 77, 105, 126, 166, 194 (100), 253, 332, 404; HRMS (M$^+$) calcd 404.1412, found 404.1401. Consistent with previous work,[6] anhydride **3** was extremely resistent to base hydrolysis (e.g. refluxing 1 N aqueous KOH was without effect).

4-(Hydroxydiphenylmethyl)-*α*-phenylbenzeneacetic Acid (6). To 4.0 mL of concentrated sulfuric acid preheated to 45 °C was added all at once the purified powdered diacid **1** (0.47 g, 1.11 mmol). Vigorous foaming immediately occurs, and the reaction mixture turns deep brownish-red. The reaction mixture was maintained at 45 °C for 7 min by which time the foaming had ceased. The reaction was quenched with ca. 10 g of crushed ice, thereby affording a grayish "milky" suspension. Extraction with four 15-mL portions of EtOAc was followed by washing the combined organic layers with three 20-mL portions of water and one 20-mL portion of saturated brine. After drying of the organic layer, the volatiles were removed to give a yellow-green glass. Purification by prep TLC (developed twice, 30% EtOAc/70% ligroin, elution of main band with EtOAc) gave a yellow oil that slowly solidified. Recrystallization from chloroform/ligroin provides analytically pure material as a faintly yellow solid (0.234 g, 0.58 mmol, 52%, mp 141–142 °C): IR ($CHCl_3$) 3800–2300 (br), 1712, 1600, 1495, 1305, 1160 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.32–7.21 (m, 19 H), 5.03 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 179.0, 146.8, 145.9, 137.7, 136.8, 128.6, 128.3, 128.2, 127.9, 127.8, 127.5, 127.2, 82.3, 57.2; MS (EI, 70 eV) *m/z* 83, 105, 126, 167, 241, 317 (100), 332, 394; HRMS (M$^+$) calcd 394.1569, found 394.1559. The solid analyzes as a semihydrate. Anal. Calcd for $(C_{27}H_{22}O_3)_2 \cdot H_2O$: C, 80.38; H, 5.74. Found: C, 80.64; H, 5.51.

Head-Space Experiments. A 25-mL Erlenmeyer flask equipped with a septum and containing 3 mL of preheated (45 °C) concentrated sulfuric acid was carefully flushed with argon. The flask was momentarily opened, and solid diacid **1** was added all at once. While the effervescing solution cooled to rt, the head space was perdiodically sampled via gas syringe and the vapor aliquots were injected into a GC/MS. A control experiment was performed using an identical apparatus and reaction medium but without the addition of the diacid (the control was sampled after ca. 20 min). Despite considerable imprecision in gas evolution kinetics (due to the increasing viscosity of the cooling reaction medium within the closed container), the relative abundance of the *m/z* 28 peak obviously increases with time (see Table I).

Methyl 4-(Hydroxydiphenylmethyl)-*α*-phenylbenzeneacetate (7). An ethereal diazomethane solution (ca. 1.2 mmol) was added to a sample of hydroxy acid **6** (0.058 g, 0.144 mmol) in methylene chloride (5 mL) followed by standing at rt for 15 h. Removal of volatiles gave a yellow oil (0.050 g). A portion (0.045 g of the crude) was purified by prep TLC (one development, 30% EtOAc/70% ligroin, elution of the main band by EtOAc). The resulting faintly yellow oil (0.034 g; 76% recovery) was analytically pure: IR ($CDCl_3$) 3596, 2928, 1734, 1492, 1154, 1009, 908 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.31–7.23 (m, 19 H), 5.01 (s, 1 H), 3.72 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 172.9, 146.7, 145.9, 138.4, 137.6, 128.6, 128.2, 128.1, 127.9, 127.3, 127.2, 81.8, 56.7, 52.3; MS (EI, 70 eV) *m/z* 57, 77, 105 (100), 149, 167, 331, 408; HRMS (M$^+$) calcd 408.1725, found 408.1757.

4-(Chlorodiphenylmethyl)-*α*-phenylbenzeneacetyl Chloride (8). Hydroxy acid **6** (0.090 g, 0.22 mmol) and oxayl chloride (0.119 mL, 1.37 mmol) were combined in benzene (10 mL), and then the resulting solution was heated at reflux under nitrogen for 0.7 h. After removal of volatiles, the crude yellow-brown oil (ca. 0.12 g) was used as rapidly as possible. An IR of the crude reaction mixture did not show OH absorption: IR (neat) 1799, 1660, 1598, 1494, 1479, 1445 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.50–7.20 (m, 19 H), 5.44 (s, 1 H); MS (EI, 70 eV) *m/z* no M$^+$ but [M$^+$ – Cl] = 395, 397 and [M$^+$ – 2 Cl] = 360.

Methyl 4-(Methoxydiphenylmethyl)-*α*-phenylbenzeneacetate (9). Freshly made acid chloride **8** (0.058 g, 0.134 mmol) was dissolved in 2.0 mL of anhydrous THF. To this was added 0.60 mL of pyridine and 2.50 mL of reagent-grade methanol. The reaction mixture was stirred at room temperature for 14 h. The volatiles were then removed. The syrupy residue was acidified with concentrated HCl and extracted with three 10-mL portions of EtOAc. The combined organic layers were washed once with saturated sodium bicarbonate solution and once with saturated brine, dried, and filtered, and the volatiles were removed to afford a residue (0.045 g) that was purified by prep TLC (development with ligroin and EtOAc (70:30), elution with EtOAc) to give a pale yellow oil (0.039 g, 69%): IR ($CHCl_3$) 1736, 1601, 1490, 1446 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.42–7.22 (m, 19 H), 4.99 (s, 1 H), 3.71 (s, 3 H), 3.02 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 172.9, 143.8, 138.5, 137.1, 128.9, 128.8, 128.6, 128.0, 127.9, 127.8, 127.3, 127.0, 86.9, 56.7, 52.3, 52.1; MS (EI, 70 eV) *m/z* 43 (100), 57, 77, 105, 273, 345, 391, 422; HRMS (M$^+$) calcd 422.1882, found 422.1878.

Ethyl 4-(Ethoxydiphenylmethyl)-*α*-phenylbenzeneacetate (10). Acid chloride **8** (0.095 g, 0.22 mmol), THF (6.0 mL), pyridine (0.81 mL), and 2.0 mL of absolute ethanol were reacted together and then subjected to workup and prep TLC purification (only one significant band) as before to afford a yellow oil (0.047 g, 47%): IR (neat) 2900, 1732, 1600, 1493, 1448, 1371, 1196, 1155, 1069, 1022 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.43–7.21 (m, 19 H), 4.97 (s, 1 H), 4.19 (q, J = 7.2 Hz, 2 H), 3.07 (q, J = 6.8 Hz, 2 H), ca. 1.23 (overlapping triplets, J = 6.8 and J = 7.2 Hz, 6 H); ^{13}C NMR ($CDCl_3$) δ 172.4, 144.3, 143.3, 138.6, 137.1, 128.6, 128.5, 127.8, 127.6, 127.2, 126.8, 61.1, 59.4, 56.7, 15.2, 14.1; MS (EI, 70 eV) *m/z* 105, 165, 188, 253, 287, 332, 359, 404 (100), 450; HRMS (M$^+$) calcd 450.2195, found 450.2193.

Direct Reaction of Acid Chloride with Ethanol in the Absence of Pyridine. Formation of both Ethyl 4-(Ethoxydiphenylmethyl)-*α*-phenylbenzeneacetate (10) and Ethyl 4-(Hydroxydiphenylmethyl)-*α*-phenylbenzeneacetate (11). Acid chloride **8** (0.058 g, 0.134 mmol), THF (2.0 mL), and ethanol (2.0 mL) were reacted together for 15 h. The residue was diluted with water (10 mL, resulting pH near 1) and extracted with three 10-mL portions of chloroform. The combined organic layers were washed with saturated sodium bicarbonate solution (10 mL) and with saturated brine (10 mL), dried, and filtered, and the volatiles were removed. Prep TLC (two developments with ligroin and EtOAc (70:30), elution with EtOAc) gave two bands (upper band was the ether–ester **10** identical to previously prepared material (0.017 g, 28%), and the lower band was the hydroxy ester **11** (0.027 g, 48%)). For hydroxy ester **11**: IR ($CHCl_3$) 3508, 2980, 1735, 1494, 1307, 1152, 1022, 699 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.31–7.21 (m, 19 H), 4.97 (s, 1 H), 4.17 (q, J = 6.9 Hz, 2 H), 2.78 (br s, 1 H), 1.23 (t, J = 6.9 Hz, 3 H); ^{13}C NMR δ 172.4, 146.7, 145.7, 138.6, 137.7, 128.5, 128.1, 127.8, 127.2, 81.8, 61.2, 56.8, 14.1; MS (EI, 70 eV) *m/z* 77, 91, 105 (100), 135, 165, 183, 253, 345, 404, 422; HRMS (M$^+$) calcd 422.1882, found 422.1906.

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Supplementary Material Available: ^{1}H NMR, ^{13}C NMR, and MS spectra for **1**, **3**, **5**, and **6–11** (23 pages). Ordering information is given on any current masthead page.