(Fellowship To A.B.) is gratefully acknowledged.

Registry No. *p*-Nitrobenzonitrile oxide, 2574-03-0; *p*-methoxybenzonitrile oxide, 15500-73-9; propene, 115-07-1; 1-butene, 106-98-9; 1-pentene, 109-67-1; 1-hexene, 592-41-6; 3-methyl-1-butene, 563-45-1; 3,3-dimethyl-1-butene, 558-37-2; isobutene, 115-11-7; 5-methyl-3-(*p*-nitrophenyl)isoxazoline, 63008-35-5; 5-ethyl-3-(*p*-nitrophenyl)-isoxazoline, 70416-55-6; 3-(*p*-nitrophenyl)-5-propylisoxazoline, 70416-56-7; 5-butyl-3-(*p*-nitrophenyl)isoxazoline, 70416-57-8; 5-isopropyl-3-(*p*-nitrophenyl)isoxazoline, 70416-58-9; 5-*tert*-butyl-3-(*p*-nitrophenyl)isoxazoline, 70428-87-4; 5,5-dimethyl-3-(*p*-nitrophenyl)isoxazoline, 70416-59-0; 3-(*p*-methoxyphenyl)-5-methylisoxazoline, 63008-36-6; 5-ethyl-3-(*p*-methoxyphenyl)isoxazoline, 70416-60-3; 3-(*p*-methoxyphenyl)-5-propylisoxazoline, 70416-61-4; 5-butyl-3-(*p*-methoxyphenyl)isoxazoline, 70416-62-5; 5-isopropyl-3-(*p*-methoxyphenyl)isoxazoline, 70416-63-6; 5-*tert*-butyl-3-(*p*-methoxyphenyl)isoxazoline, 70416-64-7; 5,5-dimethyl-3-(*p*-methoxyphenyl)isoxazoline, 70428-86-3.

Cyclobutenecarboxylic Esters via Aluminum Chloride Induced [2 + 2] Cycloadditions of 2-Propynoic Esters to Cyclic Olefins

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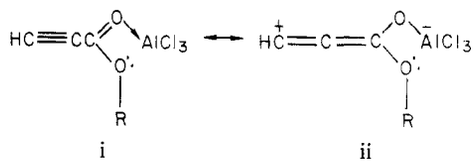
Thermal [2 + 2] cycloadditions involving unactivated olefins are quite rare.[1] Snider has demonstrated recently that 2-propynoic esters (propiolic esters)[2a,b] and also 3-butyn-2-one[2c] can be activated by Lewis acids[3] to yield [2 + 2] cycloadducts with simple alkenes. Specifically, he showed that *cis*-2-butene as well as *trans*-2-butene maintain their configuration,[2a] each giving a single cyclobutenecarboxylic ester (Scheme I).

As this subject still seems at the stage where the collection of further data is desirable, we have studied $AlCl_3$-promoted [2 + 2] cycloadditions of 2-propynoic esters (**1a,b**) to a number of cyclic alkenes (Table I). In all cases, the cycloaddition is preparatively useful as suggested,[2] proceeding over several hours or days at room temperature. The reaction with *trans*-cyclooctene is quite fast, taking a few minutes and giving adduct **5b** without discernible leakage into cis-fused **3b**. Of the two esters, the ethyl ester **1b** seems superior to the methyl ester **1a** in reacting more quickly and giving products which can be separated more easily by GC because of their reduced volatility. Mechanistically, two paths have been considered: a two-step reaction via ionic intermediate(s) with retention of olefin configuration and a concerted $_\pi2_s + _\pi2_a$ process.[2a] We suggest that complexed 2-propynoic ester is a species which has the character of an electron-deficient acetylene i and also a sterically accessible vinyl (allenyl) cation ii. In accord with the description as i, cyclopentadiene and norbornadiene furnish Diels–Alder and homo-Diels–Alder adducts **9a** and **8a**, respectively, at very much enhanced rates as expected. Note, however, that norbornadiene gives not only **8a** but also [2 + 2] adduct **7a**. We have checked independently that adduct **7a** is not formed at all in the *absence* of $AlCl_3$. Further, the pattern of reactivity of monoolefinic reaction partners in Table I together with that in the literature[2] parallels in many ways the one established by Ghosez[4] for cycloadditions of dichloroketene. In conclusion, complexed 2-propynoic ester seems capable of reacting as a conventional, but strongly activated, dienophile (cf. i) and also as a powerful ketene-like $_\pi2_a$ component (cf. ii), depending on the reaction partner.[5]



i ii

Experimental Section

All boiling points are uncorrected. Infrared spectra (CCl_4) were recorded on Perkin-Elmer 457 and 580 infrared spectrometers. NMR spectra (CCl_4) were determined on Varian EM 360 and Bruker HX 90 spectrometers (Me_4Si internal standard). The mass spectra were obtained with a Varian CH 5 (70 eV) mass spectrometer. Preparative GC was carried out on a Varian Aerograph Autoprep Model 700 (column: 6 m, 3% QF1; carrier gas hydrogen). Microanalyses are due to Frau Jirotkova of the Institut für Organische Chemie.

Cycloadditions with 2-Propynoic Esters.[2a] General Procedure. Commercial aluminum trichloride was vigorously stirred into a solution of 2-propynoic ester in dried benzene. After 15 min, evolution of heat had subsided, and the cyclic olefin was added to the resulting clear solution which was stirred at room temperature. The progress of the reaction was monitored by pouring aliquots of the reaction solution into aqueous $NaHCO_3$/ether, shaking the solution, and examining the ether layer by GC. When the product peak no longer increased substantially, the reaction mixture was poured into aqueous $NaHCO_3$/ether with precipitation of aluminum hydroxide, and the product was extracted continuously overnight with a rotatory perforator. The organic phase was washed with water until neutral and dried ($MgSO_4$), and the solvent was evaporated.

Methyl *cis*-Bicyclo[3.2.0]hept-6-ene-6-carboxylate (2a). Methyl 2-propynoate (0.84 g, 10 mmol), cyclopentene (1.36 g, 20 mmol), and aluminum trichloride (0.67 g, 5 mmol) were allowed to react in 25 mL of dried benzene according to the general procedure. Workup after 72 h afforded 0.63 g of the crude product which was distilled (Kugelrohr) to give 0.27 g (18%) of pure **2a**: bp 120 °C (10 mm); ^1H NMR[6] δ 1.0–2.0 (m, 6 H, CH_2), 3.08 (m, 1 H, CH), 3.34 (m, 1 H, CH), 3.66 (s, 3 H, OMe), 6.52 (m, 1 H, HC=C); IR ($CHCl_3$) 2955, 2860, 1712, 1610, 1600, 1436, 1277, 1135 cm^{-1}; mass spectrum, *m/e* 152 (M^+). Anal. Calcd for $C_9H_{12}O_2$ (152.2): C, 71.03; H, 7.95. Found: C, 70.82; H, 7.99.

Ethyl *cis*-Bicyclo[6.2.0]dec-9-ene-9-carboxylate (3b). Following the general procedure, ethyl 2-propynoate (2.96 g, 30 mmol), *cis*-cyclooctene (6.60 g, 60 mmol), and aluminum trichloride (2.01 g, 15 mmol) were allowed to react in 45 mL of benzene (48 h). Fractional distillation (Kugelrohr) yielded **3b** (2.7 g, 43%): bp 120 °C (0.1 mm); ^1H NMR δ 1.27 (t, J = 7 Hz, 3 H, CH_3),

(1) (a) D. Seebach in "Houben-Weyl", Vol. 4, Thieme, Stuttgart, 1971; (b) R. Huisgen, R. Grashey, and J. Sauer in "The Chemistry of Alkenes", S. Patai, Ed., Interscience, London, 1964, Chapter 11; (c) J. D. Roberts and C. M. Sharts, *Org. React.*, **12**, 1 (1962); cf. also K. Alder, unpublished work cited by M. Günzel and W. Günzel, *Angew. Chem.*, **72**, 219 (1960).

(2) (a) B. B. Snider, *J. Org. Chem.*, **41**, 3061 (1976); (b) B. B. Snider and N. J. Hrib, *Tetrahedron Lett.*, 1725 (1977); (c) B. B. Snider, L. A. Brown, R. S. Eichen Conn, and T. A. Killinger, *ibid.*, 2831 (1977).

(3) Another substituted acetylene, namely 1,2-dicyanoacetylene, shows a striking activation by $AlCl_3$. Diels–Alder addition to benzene: E. Ciganek, *Tetrahedron Lett.*, 3321 (1967). [2 + 2] cycloaddition to tetramethylthiophene: H. Wynberg and R. Helder, *ibid.*, 3647 (1972); cf. also R. Helder and H. Wynberg, *ibid.*, 605 (1972).

(4) (a) R. Montaigne and L. Ghosez, *Angew. Chem., Int. Ed. Engl.*, **7**, 221 (1968); (b) L. Ghosez, R. Montaigne, A. Roussel, H. Vanlierde, and P. Mollet, *Tetrahedron*, **27**, 615 (1971); (c) L. Ghosez and M. J. O'Donnell, *Pericyclic React.*, **2**, 79 (1977). See also L. R. Krespi and A. Hassner, *J. Org. Chem.*, **43**, 2879 (1978); W. T. Brady, *Synthesis*, 415 (1971); H. C. Stevens, D. A. Reich, D. R. Brandt, K. R. Fountain, and E. J. Gaughan, *J. Am. Chem. Soc.*, **87**, 5257 (1965).

(5) [2 + 2] cycloadditions of acetylenic esters to electron-rich olefins such as enamines proceed presumably stepwise; see D. N. Reinhoudt, J. Geevers, and W. P. Trompenaars, *Tetrahedron Lett.*, 1351 (1978), and literature cited.

(6) Cf. L. A. Paquette and G. Zon, *J. Am. Chem. Soc.*, **96**, 224 (1974).

0022-3263/79/1944-2802$01.00/0

Scheme I

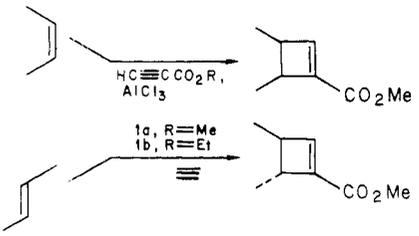
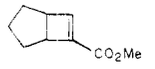
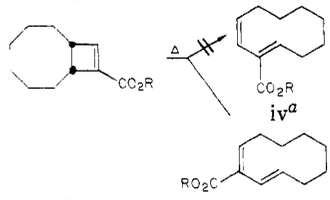
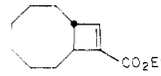
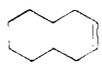
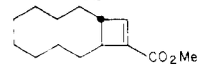
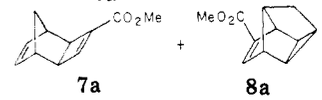
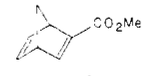


Table I. Cycloadducts from the $AlCl_3$-Catalyzed Reaction of 2-Propynoic Esters (1a, R = Me; 1b, R = Et) with Cyclic Olefins

cyclic olefin	cycloadduct	isolated yield, %
	 2a	18
	 3a, R = Me; b, R = Et; 4a, b; iv[a]	40; 43
	 5b	63
	 6a	19
	 7a + 8a	33[c]
	 9a[b]	70

[a] Interestingly, isomer iv with the more crowded trisubstituted double bond was not formed. [b] A [2 + 2] adduct was not formed at all (GC). [c] 7a:8a is ~2:1.

1.2–2.3 (m, 12 H, CH_2), 2.66 (m, 1 H, CH), 2.90 (m, 1 H, CH), 4.21 (q, J = 7 Hz, 2 H, OCH_2), 6.55 (d, J = 1.1 Hz, 1 H, HC=C); IR 3055, 2920, 1715, 1619, 1609 cm^{-1}; mass spectrum, m/e 208 (M^+). Anal. Calcd for $C_{13}H_{20}O_2$ (208.3): C, 74.96; H, 9.68. Found: C, 75.01; H, 9.75.

Methyl (*E*,*E*)-1,9-Cyclodecadiene-1-carboxylate (4a). Methyl 2-propynoate (4.2 g, 50 mmol), *cis*-cyclooctene (11.0 g, 100 mmol), and aluminum trichloride (3.4 g, 25 mmol) were allowed to react in 75 mL of benzene under the usual conditions (23 h). The crude product (8.3 g) was distilled (Kugelrohr) to afford 4.3 g of *cis*-cyclooctene and 3.9 g (40%) of a colorless oil, bp 120 °C (0.1 mm), which on the basis of its ^1H NMR spectrum consisted mainly of **3a**. On preparative GC (column temperature 185 °C), 0.8 g of the oil gave a minor fraction (35 mg) of an isomeric mixture [m/e 194 (M^+)], which was not identified further, and then **4a**[7] (0.30 g after Kugelrohr distillation): ^1H NMR δ 1.39 (m, 8 H, CH_2), 2.19 (m, 4 H, CH_2), 3.68 (s, 3 H, OCH_3), 5.43 (d of t, J = 16, 7 Hz, 1 H, HC=C), 5.93 (br d, J = 16 Hz, 1 H, HC=C), 6.76 (d of t, J = 1.5, 8 Hz, 1 H, HC=CCO_2R); IR 2930, 2855, 1706, 1653, 1606, 1458, 1435, 1264, 978 cm^{-1}; mass spectrum, m/e 194 (M^+). Anal. Calcd for $C_{12}H_{18}O_2$ (194.3): C, 74.19; H, 9.34. Found: C, 74.20; H, 9.35.

Ethyl *trans*-Bicyclo[6.2.0]dec-9-ene-9-carboxylate (5b).[8] Ethyl 2-propynoate (0.98 g, 10 mmol) and aluminum trichloride (0.67 g, 5 mmol) in 15 mL of benzene were allowed to react with *trans*-cyclooctene[9] (2.20 g, 20 mmol) according to the general procedure. The temperature in the flask immediately rose to 50 °C. After a few minutes, most of the cycloadduct had been formed (GC). The solution was stirred (1 h) and worked up by fractional distillation (Kugelrohr) to afford **5b** (1.3 g, 63%) as a colorless oil: bp 105 °C (0.1 mm); ^1H NMR δ 1.28 (t, J = 7 Hz, 3 H, CH_3), 1.0–2.7 (m, 14 H, CH_2 and CH), 4.11 (q, J = 7 Hz, 2 H, OCH_2), 6.63 (m, 1 H, HC=C); IR 3055, 2991, 2925, 2853, 1716, 1610, 1478, 1454, 1258, 1134, 1119, 1052, 861 cm^{-1}; mass spectrum, m/e 208 (M^+). Anal. Calcd for $C_{13}H_{20}O_2$ (208.3): C, 74.96; H, 9.68. Found: C, 74.89; H, 9.82.

Methyl *cis*-Bicyclo[8.2.0]dodec-11-ene-11-carboxylate (6a). Methyl 2-propynoate (2.56 g, 30 mmol), *cis*-cyclodecene (8.28 g, 60 mmol), and aluminum trichloride (2.01 g, 15 mmol) were allowed to react in 45 mL of benzene as described above. After 7 days, the reaction was stopped, and the solvent was evaporated to give 8.1 g of a yellow liquid, which afforded 5.3 g of *cis*-cyclodecene and **6a** (1.3 g, 19%), bp 140 °C (0.1 mm) (Kugelrohr) as a clear liquid: ^1H NMR δ 1.1–2.1 (m, 16 H, CH_2), 2.78 (m, 1 H, CH), 3.02 (m, 1 H, CH), 3.66 (s, 3 H, OCH_3), 6.56 (d, J = 1.3 Hz, 1 H, HC=C); IR 3050, 2925, 2865, 2855, 1721, 1611, 1475, 1443, 1434, 1140 cm^{-1}; mass spectrum, m/e 222 (M^+). Anal. Calcd for $C_{14}H_{22}O_2$ (222.3): C, 75.63; H, 9.97. Found: C, 75.32; H, 9.98.

Reaction of Norbornadiene and Methyl 2-Propynoate. Methyl 2-propynoate (4.2 g, 50 mmol), norbornadiene (9.2 g, 100 mmol), and aluminum trichloride (3.4 g, 25 mmol) were stirred for 47 h in 75 mL of benzene. On Kugelrohr distillation (bp 110 °C (0.1 mm)), 2.9 g (33%) of an isomeric mixture [**7a**:**8a** ~ 2:1] was isolated. Preparative GC (column temperature 160 °C) on 1 g of the mixture afforded 0.33 g of **methyl *exo*-tricyclo[4.2.1.$0^{2,5}$]nona-3,7-diene-3-carboxylate (7a)**: ^1H NMR[10] δ 1.38 (m, 2 H, CH_2), 2.34 (m, 1 H, CH), 2.48 (m, 1 H, CH), 2.63 (m, 2 H, 2CH), 3.70 (s, 3 H, OCH_3), 6.14 (m, 2 H, HC=CH), 6.89 (s, 1 H, HC=CCO_2R); IR[9] 3060, 1720, 1605, 1595, 1562, 1123, 702 cm^{-1}; mass spectrum, m/e 176 (M^+).

As a second fraction 0.11 g of **methyl tetracyclo[4.3.0.$0^{2,4}$.$0^{3,7}$]non-8-ene-carboxylate (8a)** was isolated: ^1H NMR[11] δ 1.40 (m, H-2, H-3), 1.61 (m, 2 H, CH_2), 1.81 (m, H-4), 2.11 (m, H-6), 2.71 (m, H-1), 2.99 (m, H-7), 3.68 (s, 3 H, OCH_3), 6.87 (d, J = 3 Hz, 1 H, HC=C); IR[10] 3060, 1718, 1590, 1240, 1089 cm^{-1}; mass spectrum, m/e 176 (M^+). Anal. Calcd for $C_{11}H_{12}O_2$ (176.2): C, 74.98; H, 6.86. Found: C, 74.63; H, 6.83.

Homo-Diels–Alder Addition in the Absence of Aluminum Trichloride. Methyl 2-propynoate (1.26 g, 15 mmol) and norbornadiene (4.6 g, 50 mmol) were refluxed for 60 h to give **8a**[10] (5%) without any **7a**.

Methyl Bicyclo[2.2.1]hepta-2,5-diene-2-carboxylate (9a). Methyl 2-propynoate (0.84 g, 10 mmol), cyclopentadiene (0.66 g, 10 mmol), and aluminum trichloride (0.67 g, 5 mmol) were allowed to react in 15 mL of benzene. An exothermic reaction ensued. After the solution was stirred for 15 min, the Diels–Alder adduct **9**[12] was obtained (1.05 g, 70%) without any [2 + 2] adduct (GC).

Acknowledgments. We thank the Fonds der Chemischen Industrie for support of our work.

Note Added in Proof: After this paper had been submitted, further [2 + 2] cycloadditions of 2-propynoic esters to activated and unactivated alkenes were described: see R. D. Clark and K. G. Untch, *J. Org. Chem.*, **44**, 248 (1979); B. B. Snider and D. M. Roush, *J. Am. Chem. Soc.*, **101**, 1906 (1979).

(7) Interestingly, the cycloadduct from cyclododecene and methyl 2-propynoate suffers controtatory opening at 25 °C; see ref 2b.

(8) The parent hydrocarbon has been described by P. Radlick, R. Klem, S. Spurlock, J. J. Sims, E. E. van Tamelen, and T. Whitesides, *Tetrahedron Lett.*, 5117 (1968).

(9) J. N. Hines, M. J. Peagram, E. J. Thomas, and G. H. Whitham, *J. Chem. Soc., Perkin Trans. 1*, 2332 (1973).

(10) Cf. W. Eberbach, *Chem. Ber.*, **107**, 3287 (1974).

(11) Cf. H. Prinzbach and D. Hunkler, *Chem. Ber.*, **106**, 1804 (1973).

(12) H. Prinzbach and M. Thyes, *Chem. Ber.*, **104**, 2489 (1971).

Registry No. **1a**, 922-67-8; **1b**, 623-47-2; **2a**, 51326-24-0; **3a**, 70288-54-9; **3b**, 70288-55-0; **4a**, 70288-56-1; **4b**, 70288-57-2; **5b**, 70288-58-3; **6a**, 70288-59-4; **7a**, 13155-85-6; **8a**, 42132-17-2; **9a**, 3604-36-2; *cis*-cyclooctene, 931-87-3; *trans*-cyclooctene, 931-89-5; *cis*-cyclodecene, 935-31-9; norboradiene, 121-46-0; cyclopentadiene, 542-92-7; cyclopentene, 142-29-0; aluminum chloride, 7446-70-0.

Synthesis of [5.1]Metacyclophane

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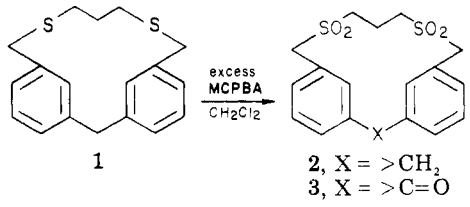
Received February 9, 1979

In an earlier publication[1] we mentioned the synthesis of [5.1]metacyclophane **10**, which we now describe. Vögtle[2] recently published the first syntheses of both [4.1]- and [5.1]metacyclophane by use of the sulfone pyrolysis method.

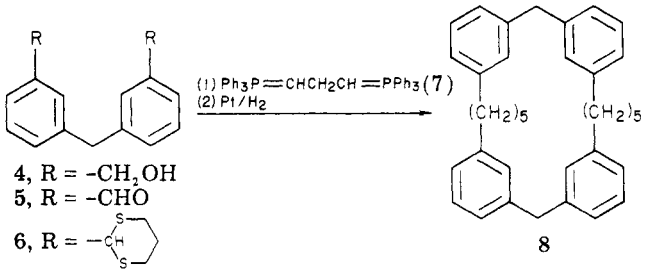
Syntheses of [*n*.1]metacyclophanes have not been commonplace due to the lack of a general method such as that developed by Boekelheide for the [2.2]metacyclophanes.[3] However, while the sulfone pyrolysis procedure developed by Vögtle[2] and Staab[4] promises to meet the requirement of general applicability, it appears to be limited with regard to the amounts of cyclophanes which can be produced. The bis(sulfone) **2** was available to us by oxidation, with excess peracid, of the bis(sulfide) **1** (see Scheme I). Despite the structural similarity to the bis(sulfone) **3** used successfully by Vögtle,[2] we were unable to induce thermal cracking of the bis(sulfone) **2** in a preparatively useful manner. Other routes suitable for the preparation of multigram quantities of [5.1]metacyclophane **10** were therefore explored.

As quite strained olefins had been synthesized by intramolecular Wittig reactions,[5] this reaction was the first approach we considered. More particularly, bis Wittig reactions had been widely used to prepare other types of macrocyclic compounds.[6] The dialdehyde **5** was readily prepared by Jones oxidation from the previously described bis(carbinol) **4**.[1] Reaction of this dialdehyde **5** with the bis(ylide) **7** obtained from 1,3-trimethylenebis(triphenylphosphonium) bromide[7] and 2 equiv of butyllithium yielded, after catalytic reduction of the product, not the desired product of intramolecular bridging, i.e., the [5.1]metacyclophane **10**, but the intermolecularly bridged dimeric product **8**. The structure of this product was evident by the strong molecular ion peak in the mass spectrum at *m/e* 472 (see Scheme II).

Other methods of inserting the three-carbon bridge were considered. The bis(dithiane) **6** could be readily prepared from the dialdehyde **5**. Although attempts to react the dilithio derivative of the bis(dithiane) **6** with a variety of 1,3-dihalopropanes were unsuccessful, a stepwise procedure was successful in providing the bridged dithiane **9**. Reaction of the lithio derivative with 1,3-bromochloropropane yielded the 3-chloropropyl dithiane contaminated with traces of starting material and presumably the dialkylated product. The crude monoalkylated material was treated

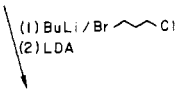


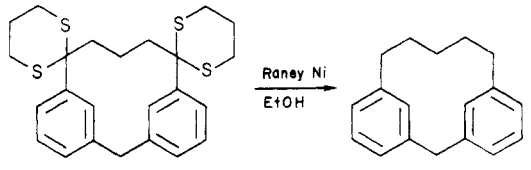
1 2, X = $>CH_2$ 3, X = $>C=O$



4, R = $-CH_2OH$
5, R = $-CHO$
6, R =

8





9 10

with lithium diisopropylamide (LDA) in THF. The crystalline-bridged dithiane **9** could be isolated by filtration through neutral III alumina. The overall yield of **9** for the two steps from the dithiane **6** was 30%, which, for the formation of this type of macrocycle, was considered satisfactory. Desulfurization of the bridged dithiane **9** with Raney nickel yielded [5.1]metacyclophane **10**, which was purified by preparative GLC and crystallized from methanol/pentane to yield a product with mp 53–54 °C. The spectral data obtained were similar to those reported by Vögtle,[2] but a direct comparison of the samples or spectra was not made.[8]

The yield on the desulfurization step was surprisingly poor (15%), but no attempt was made to study this process or employ other methods of removing dithioketals and then reducing the resulting diketone to **10**. The process though lends itself well to scale up and promises to be a route by which multigram quantities of [5.1]metacyclophane **10** can be obtained.

Experimental Section

General Procedures. Melting points were determined in a Thomas-Hoover melting point apparatus and are uncorrected. NMR spectra were obtained on a Varian A60 spectrometer in $CDCl_3$, unless otherwise stated, infrared spectra on a Perkin-Elmer 21 or 521 spectrophotometer, mass spectra on an AEI MS902 spectrometer at 70 eV, and ultraviolet spectra on a Cary 14 instrument.

2,6-Dithia[7.1]metacyclophane 2,2,6,6-Tetraoxide (2). 2,6-Dithia[7.1]metacyclophane[1] (2 g, 0.0067 mol) was dissolved in CH_2Cl_2 (50 mL). With stirring, *m*-chloroperbenzoic acid (5 g, 0.0286 mol) was added portionwise during 30 min, which caused a gentle reflux. As the mixture cooled, a solid separated, which was collected. The filtrate was washed (10% aqueous $KHCO_3$) and dried ($MgSO_4$), and the solvent was removed. The residue

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(3) Boekelheide, V.; Anderson, P. H.; Hylton, T. A. *J. Am. Chem. Soc.* **1974**, *96*, 1558–64.
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(5) Becker, K. B. *Chimia* **1974**, *28*, 726–7.
(6) Vollhardt, K. P. C. *Synthesis* **1975**, 765–80.
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(8) Professor Vögtle did not respond to our letter or comment on the 100-MHz spectrum of **10** contained therein.

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