Studies in Furan Chemistry. VII.[1] Synthesis of Four Isomeric 7,8:17,18-Dibenzo[20]annulene 1,4:11,14-Dioxides

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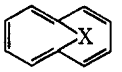
Received October 30, 1967

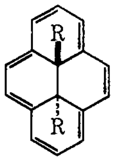
Abstract: Three isomeric 7,8:17,18-dibenzo[20]annulene 1,4:11,14-dioxides (V, VI, and VII) have been synthesized by treatment of equimolar portions of 2,5-furandialdehyde and *o*-xylylenebis(triphenylphosphonium bromide) with lithium ethoxide. Irradiation of V, VI, or VII with sunlight led to a fourth, all-*trans* isomer, VIII. The properties of these four isomers are reported.

The observed properties of the large variety of annulenes and dehydroannulenes synthesized by Sondheimer and his coworkers[2] in recent years have substantiated the predictions of Hückel's rule.[3] A number of aromatic anions[4] and cations[4] containing $(4n + 2)$ π electrons have also been prepared.

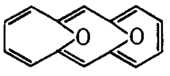
If pairs of internal hydrogen atoms in the annulenes are replaced by one-atom bridges, the peripheral conjugation is not perturbed significantly and those systems containing $(4n + 2)$ peripheral π electrons still exhibit aromaticity. A number of such bridge-bonded derivatives of [10]annulene (I)[5] and [14]annulene (II[6] and III[7]) have now been synthesized and the experimental evidence confirms that all these compounds are aromatic A variety of bridge-bonded [18]annulenes have also been synthesized,[8] and if the heteroatoms are such that a planar or near-planer configuration is possible (IV, X = O, S) they exhibit peripheral delocalization rather than localized aromaticity confined to the component furan or thiophene nuclei.



I, X = CH_2, O, NH, NCH_3, $NCOCH_3$.



II, R = Me, Et



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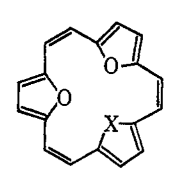
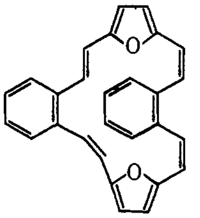


IV

We now report the synthesis of the isomeric hetero-bridged [20]annulene derivatives, 7,8:17,18-dibenzo-[20]annulene 1,4:11,14-dioxide (V–VIII).



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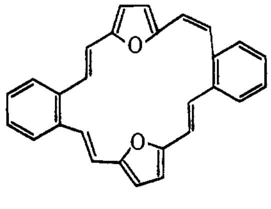

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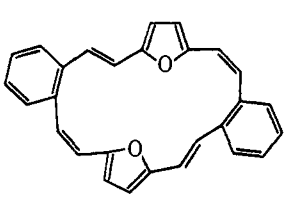
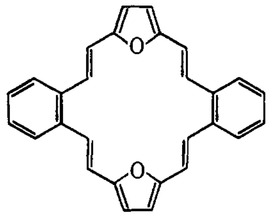


VII



VIII

The reaction of 2,5-furandialdehyde (IX)[9] with *o*-xylylenebis(triphenylphosphonium bromide) (X)[10] on addition of lithium ethoxide led to a variety of products including a mixture of three isomeric 7,8:17,18-di-

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(1) For paper VI see J. A. Elix and M. V. Sargent, *J. Chem. Soc., C*, in press.

(2) See F. Sondheimer, *Pure Appl. Chem.*, 7, 363 (1963); F. Sondheimer, *Proc. Roy. Soc.* (London), **A297**, 173 (1967); F. Sondheimer, I. C. Calder, J. A. Elix, Y. Gaoni, P. J. Garratt, K. Grohmann, G. Di Maio, J. Mayer, M. V. Sargent, and R. Wolovsky, Special Publication No. 21, The Chemical Society, London, 1967, p 75.

(3) E. Huckel, *Z. Phys. Chem.*, **70**, 204 (1931); **72**, 310 (1931); **76**, 628 (1932); "Grundzüge der Theorie ungesättiger und aromatischer Verbindungen," Verlag Chemie, Berlin, 1938.

(4) See D. Ginsberg, Ed., "Non-benzenoid Aromatic Compounds," Interscience Publishers, Inc., New York, N. Y., 1959; D. Lloyd, "Carbocyclic Non-Benzenoid Aromatic Compounds," Elsevier Publishing Co., New York, N. Y., 1966; P. J. Garratt and M. V. Sargent, "Advances in Organic Chemistry, Methods and Results," Vol. 6, Interscience Publishers, Inc., New York, N. Y., in press.

(5) E. Vogel and H. D. Roth, *Angew. Chem.*, **76**, 145 (1964); *Angew. Chem. Intern. Ed. Engl.*, **3**, 228 (1964); E. Vogel, M. Biskup, W. Pretzer, and W. A. Böll, *Angew. Chem.*, **76**, 785 (1964); *Angew. Chem. Intern. Ed. Engl.*, **3**, 642 (1964); F. Sondheimer and A. Shani, *J. Am. Chem. Soc.*, **86**, 3168 (1964); E. Vogel, W. Pretzer, and W. A. Böll, *Tetrahedron Letters*, 3613 (1965).

(6) V. Boekelheide and J. B. Phillips, *J. Am. Chem. Soc.*, **85**, 1545 (1963); *Proc. Natl. Acad. Sci. U. S.*, **51**, 550 (1964); *J. Am. Chem. Soc.*, **89**, 1965 (1967); V. Boekelheide and T. Miyasaka, *ibid.*, **89**, 1709 (1967); J. B. Phillips, R. J. Molyneux, E. Sturm, and V. Boekelheide, *ibid.*, **89**, 1704 (1967).

(7) E. Vogel, M. Biskup, A. Vogel, and H. Günther, *Angew. Chem.*, **78**, 755 (1966), *Angew. Chem. Intern. Ed. Engl.*, **5**, 734 (1966); E. Vogel and H. Günther, *Angew. Chem.*, **79**, 429 (1967), *Angew. Chem. Intern. Ed. Engl.*, **6**, 385 (1967).

(8) G. M. Badger, J. A. Elix, and G. E. Lewis, *Australian J. Chem.*, **18**, 70 (1965); **19**, 1221 (1966); G. M. Badger, G. E. Lewis, and U. P. Singh, *ibid.*, **19**, 257 (1966); **19**, 1461 (1966); **20**, 1635 (1967).

(9) W. F. Cooper and W. H. Nuttall, *J. Chem. Soc.*, **101**, 1074 (1912); K. Y. Novitskii, V. P. Volkov, and Y. K. Yur'ev, *Zh. Obshch. Khim.*, **31**, 538 (1961); P. Pastour and C. Plantard, *Compt. Rend.*, **262**, 1539 (1966).

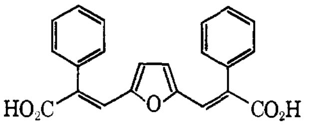
(10) C. E. Griffin, K. R. Martin and B. E. Douglas, *J. Org. Chem.*, **27**, 1627 (1962); C. E. Griffin and J. A. Peters, *ibid.*, **28**, 1715 (1963).

benzo[20]annulene 1,4:11,14-dioxides (V, VI, and VII).[11]

The three isomers were separated and purified by column chromatography followed by preparative layer chromatography.

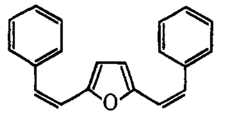
Irradiation of V, VI, or VII in chloroform solution with sunlight led to a fourth isomer VIII having the all-*trans* configuration. The photoisomerization of VI into VIII was by far the most rapid and efficient conversion, presumably because it involves isomerization about only one double bond. It was interesting to note that the properties of this all-*trans* isomer VIII differed very markedly from those of the other three isomers. Indeed, the high melting point and extremely low solubility of this compound led us to consider the possibility that VIII was in fact, a photodimer of the cyclobutane type. This surmise was repudiated by the mass spectrum (molecular ion at *m/e* 388) together with evidence that VIII sublimes unchanged at 330° (atmospheric pressure) and much more readily at reduced pressure. Cyclobutane photodimers normally undergo ready pyrolytic reversion to the olefinic monomers.[12] The structure of VIII was finally confirmed by the nmr spectrum which showed only olefinic and aromatic absorption.

In order to determine the stereochemistry and assess the extent of cyclic delocalization (if any) in the compounds V–VIII, several acyclic 2,5-di(β-phenylvinyl)-furans were synthesized for comparison. The base-catalyzed condensation of 2,5-furandialdehyde (IX) with phenylacetic acid gave *cis,cis*-α,α′-diphenyl-β,β′-(2,5-furan)diacrylic acid (XI).

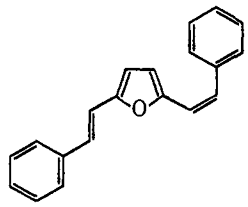


XI

The *cis,cis* configuration of XI followed from the low-field absorption of the vinylic protons in the nmr spectrum (singlet, τ 2.45) due to the deshielding by the adjacent carbonyl groups.[8,13] Decarboxylation of XI with copper chromite and quinoline at 210–220° gave a mixture of *cis,cis*-2,5-di-(β-phenylvinyl)furan (XII) and *cis,trans*-2,5-di(β-phenylvinyl)furan (XIII). The former isomer rapidly reverts to the latter, XIII, on standing at room temperature.



XII



XIII

A comparison of some relevant spectral data of the cyclic 7,8:17,18-dibenzo[20]annulene 1,4:11,14-dioxides (V–VIII) with that of the acyclic 2,5-di(β-phenylvinyl)-furans (XII and XIII) is presented in Table I. This comparison revealed (a) the probable stereochemistry of the isomers V, VI, VII, and VIII; and (b) there was little, if any, cyclic delocalization in isomers V, VI, and VII but some extended conjugation in VIII.

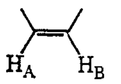
Table I. Comparative Spectral Data

	Nmr		Infrared		
Compd	*J*, cps	Proton count	*t*-CH═CH (def)	λ, mμ	ε
XII	3	4	. . .	233^a	14,300
				273	14,300
				280	13,500
				355	21,700
XIII	3	2	960	235^a	15,700
	6	2	980	243	14,600
				251	13,100
				274	22,700
				282	21,800
				366	34,900
				382	52,400
				396	33,600
V	2	2	960	206^a	36,800
	3	4	972	333	38,400
	7	2			
VI	3	2	950	208^a	33,100
	7	2	960	282	28,900
	12	2	972	325	45,300
	18	2	988	400	13,000
VII	2	2	965	208^a	43,200
	4	2	990	264	35,700
	6	2		289	40,100
	12	2		353	37,600
VIII	12	8	938	303^b	45,100
			953	314	52,300
			965	358	60,500
			990	375	84,100
Bisdehydro-[20]annulene^e				325^c	107,000
				343	76,000
[20]Annulene^e				268–272^d	1.13^f
				283	1.61^f
				297	0.87^f
				312	0.69^f
				373	0.26^f
				394	0.24^f

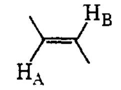
^{a} 95% ethanol. ^{b} Chloroform. ^{c} Benzene. ^{d} Pentane. ^{e} F. Sondheimer and Y. Gaoni, *J. Am. Chem. Soc.*, **84**, 3520 (1964). ^{f} Optical density.

Stereochemistry of Isomers V, VI, VII, and VIII

The allocation of configuration to these isomers was primarily based on the difference in coupling constants in the AB spectra of the *cis*- and *trans*-olefinic protons, *i.e.*



$J_{AB} = 2\text{–}4\text{cps}$



$J_{AB} = 6\text{–}18\text{cps}$

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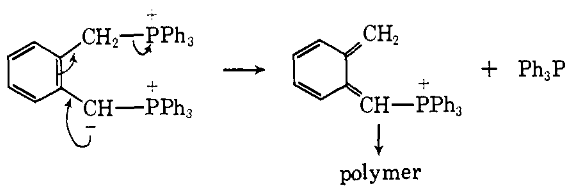
[11] Triphenylphosphine was obtained as a by-product in this reaction and is probably formed from a 1,4 elimination in the monoylide, *i.e.*



see also J. A. Elix, M. V. Sargent, and F. Sondheimer, *J. Am. Chem. Soc.*, **89**, 5080 (1967). *trans*-But-2-ene-1,4-di(triphenylphosphonium bromide) undergoes an analogous elimination (J. A. Ford and C. V. Wilson, *J. Org. Chem.*, **26**, 1433 (1961); H. Burger, Doctoral Dissertation, University of Tübigen, 1958).

[12] H. Stobbe and F. Zschoch, *Ber.*, **60**, 462 (1927); R. Stoermer and E. Asbrand, *ibid.*, **64**, 2796 (1931); M. Pailer and V. Muller, *Monatsh.*, **79**, 615 (1948); E. H. White and H. C. Dunathan, *J. Am. Chem. Soc.*, **78**, 6055 (1956); M. V. Sargent and C. J. Timmons, *J. Chem. Soc.*, 5544 (1964).

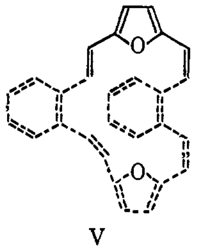
[13] L. M. Jackman and R. H. Wiley, *Proc. Chem. Soc.*, 196 (1958).

(see Table I for observed *J* values). The large range of coupling constants (6–18 cps) for the *trans*-olefinic protons probably arises from twisting of the double bonds. This makes a completely unequivocal assignment of configuration to the four isomers difficult, but even so the *J* values appear confined to two reasonably distinct groups.

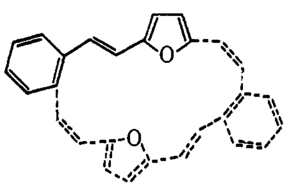
Additional evidence was provided by the infrared spectra of V–VIII. The intensity of the CH=CH (*trans*) deformation frequency (960–965 cm^{-1})[14] relative to the intensity of the furan ring breathing frequency[15] (1010–1025 cm^{-1}) was found to be proportional to the number of *trans* double bonds present (see Figure 1). This evidence substantiated the stereochemical assignment.

π-Electron Delocalization in V, VI, VII, and VIII

The nmr spectra of all isomers showed proton chemical shifts akin to those of the acyclic 2,5-di(β-phenylvinyl)furans (XII and XIII). However the chemical shifts of the olefinic protons in V, VI, VII, and VIII, are a little upfield from the reference linear compounds. This could be indicative of a weak peripheral paramagnetic ring current,[2] but is more likely to be due to special shielding effects arising from the geometry of these protons, relative to the adjacent aromatic nuclei. The fact that the cyclic isomers V, VI, and VII exhibit hypochromic effects and hypsochromic shifts in their electronic spectra relative to the acyclic 2,5-di(β-phenylvinyl)furans, indicated that there was no extended peripheral conjugation in these compounds. An obvious reason for this was revealed by a study of molecular models of V, VI, and VII. This suggested that these isomers exhibited considerable distortions from planarity and that only certain portions of the molecule could possibly participate in effective π-bond overlap, *e.g.*, in isomers V and VII below, the atoms outlined in heavy type represent the most extended, near-coplanar, conjugated systems.



V



VII

The electronic spectrum of the all-*trans* isomer VIII showed a marked hyperchromic effect relative to the other cyclic isomers and the acyclic analogs. This was indicative of some increase in conjugation which accompanied the considerable increase in rigidity and planarity of the macrocyclic system in this isomer. However it was not possible to determine the limits of this conjugation from the above data.

Experimental Section

All melting points were measured on a Kofler hot-stage apparatus. Proton magnetic resonance spectra were measured either on a Varian HA-100 or a Perkin-Elmer 60-Mcps spectrometer. The electronic spectra were determined on a Perkin-Elmer Model 137 spectrophotometer. Infrared spectra were taken on a Perkin-Elmer Model 257 grating spectrophotometer and mass spectra were measured on a A.E.I. MS-9 mass spectrometer with an inlet heated to 200°, by the direct insertion technique.

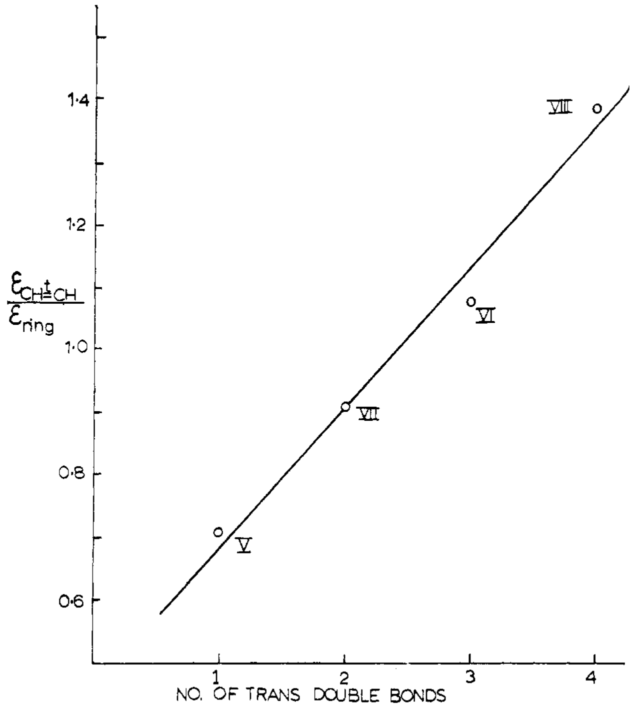


Figure 1. Plot of the relative infrared intensities of the *trans*-CH=CH deformation band against the number of *trans* double bonds (as indicated by nmr) of V, VI, VII, and VIII.

7,8:17,18-Dibenzo[20]annulene 1,41:11,14-Dioxide, Isomers V, VI, **and VII.** A mixture of *o*-xylylenebis(triphenylphosphonium bromide) (6.0 g) and 2,5-furandialdehyde (0.91 g) in anhydrous dimethylformamide (130 ml, distilled from calcium hydride) was heated to 90° in an atmosphere of nitrogen. A solution of lithium ethoxide (0.02 mol) in absolute ethanol (33 ml) was added to the reaction mixture with stirring over a period of 5 hr. The dark colored reaction mixture was allowed to cool and was diluted with cold water (500 ml). The precipitated material was extracted with ether; the ethereal extracts were washed with water, dried ($MgSO_4$), and evaporated to give a bright orange solid. This solid was extracted several times with boiling petroleum ether (bp 40–60°) and the residue discarded. Concentration of the petroleum ether extracts gave a yellow-orange solid which was adsorbed on to a column of silicic acid (45 × 2 cm, Mallinckrodt 100 mesh). The column was developed with pentane (1 l.) followed by pentane–2% ether (1 l.) and pentane–5% ether (2 l.). The first 600 ml of eluate collected contained only triphenylphosphine (0.55 g). The following 750 ml contained a mixture of triphenylphosphine, isomers V, VI, and VII and slower moving material. The solution was concentrated and the required three isomers were separated by preparative thin layer chromatography using 1.5 mm silica gel (Merck $HF_{254+366}$) plates and 2% ether–pentane as eluent. Three distinct yellow bands developed and these were removed in turn and extracted into chloroform. The chloroform extracts were concentrated separately, and the residue obtained in each case rechromatographed on a similar thin layer plate. After extraction and concentration the separate residues were recrystallized from pentane.

(a) **The 5,6-*cis*-9,10-*cis*-15,16-*cis*-19,20-*trans* isomer** V (24 mg, 1.7%) was obtained from the fastest moving yellow band and crystallized in yellow-orange prisms, mp 230–234° dec. The mass spectrum exhibited a base peak due to the molecular ion at *m/e* 388.1464 ($C_{28}H_{20}O_2$ requires *m/e* 388.1363). The nmr spectrum (100 Mcps, CCl_4) exhibited a series of peaks at τ 2.77–3.32 (8 H) due to the benzenoid protons, an AB quartet centered at 3.45, 3.71 (2 H, $J = 7$ cps) due to the *trans*-olefinic protons, singlets at 3.65 (1 H), 3.84 (2 H), and 3.90 (1 H) due to the furan ring protons, and three AB quartets centered at 3.56, 3.65 (2 H, $J = 2$ cps), 3.97, 4.11 (2 H, $J = 3$ cps), and 4.19, 4.57 (2 H, $J = 3$ cps) due to the *cis*-

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[14] L. J. Bellamy, "The Infrared Spectra of Complex Molecules," 2nd ed, John Wiley and Sons, Inc., New York, N. Y., 1959.

[15] A. R. Katritzky and J. M. Lagowski, *J. Chem. Soc.*, 657 (1959); R. Grigg, J. A. Knight, and M. V. Sargent, *ibid.*, 6057 (1965).

olefinic protons. The infrared spectrum ($CHCl_3$) of V showed prominent absorption at 3060, 3035, 3010, 1640, 1025, 972, and 960 cm^{-1}. The electronic spectrum (ethanol) exhibited maxima at 206 and 333 mμ (ϵ 36,800 and 38,400, respectively).

Anal. Calcd for $C_{28}H_{20}O_2$: C, 86.66; H, 5.16. Found: C, 86.31; H, 5.33.

(b) **The 5,6-*cis*-9,10-*trans*-15,16-*trans*-19,20-*trans* isomer** VI (28.4 mg, 2.0%) was obtained from the subsequent yellow band, and crystallized from pentane as yellow leaflets, mp 202–204° dec. The molecular ion peak at *m/e* 388.1464 was the base peak of the mass spectrum. The nmr spectrum (100 Mcps, CCl_4) showed a complex multiplet at τ 2.42–2.82 (8 H) due to the benzenoid protons, an AB quartet centered at 2.69, 3.09 (2 H, J = 18 cps), an overlapping AB quartet centered at 3.10, 3.34 (2 H, J = 12 cps), and a third AB quartet centered at 3.48, 3.69 (2 H, J = 7 cps) all due to the *trans*-olefinic protons, an AB quartet centered at 3.98, 4.24 (2 H, J = 3 cps) arising from the *cis*-olefinic protons, and two singlets at 3.40 (1 H) and 3.85 (3 H) due to the furan ring protons. The infrared spectrum of VI in $CHCl_3$ had prominent bands at 3060, 3010, 1633, 1018, 988, 972, 960, and 952 cm^{-1}. The electronic spectrum (ethanol) exhibited maxima at 208, 282, 325, and 400 mμ (ϵ 33,100, 28,900, 45,300, and 13,000, respectively).

Anal. Calcd for $C_{28}H_{20}O_2$: C, 86.66; H, 5.16. Found: C, 86.50; H, 5.38.

(c) **The 5,6-*cis*-9,10-*trans*-15,16-*cis*-19,20-*trans* isomer** VII (23 mg, 1.6%) was obtained from the third yellow band to be eluted and crystallized as yellow-orange needles, mp 209–211°. A molecular ion peak was observed at *m/e* 388.1464 in the mass spectrum. The nmr spectrum (100 Mcps, CCl_4) showed two multiplets at τ 2.52–2.66 (4 H) and 2.76–2.88 (4 H) due to the benzenoid protons, one AB quartet centered at 2.97, 3.53 (2 H, J = 6 cps), and another at 3.26, 3.56 (2 H, J = 12 cps) due to the *trans*-olefinic protons, an AB quartet centered at 3.70, 3.79 (2 H, J = 2 cps) the higher field bands of which overlap with the low-field bands of another AB quartet centered at 3.76, 3.88 (2 H, J = 4 cps) all due to the *cis*-olefinic protons, and three singlets at 3.16 (1 H), 3.69 (2 H), and 3.86 (1 H) due to the furan ring protons. The infrared spectrum ($CHCl_3$) showed prominent bands at 3060, 3015, 1633, 1020, 990, and 965 cm^{-1}. The electronic spectrum (ethanol) exhibited maxima at 208, 264, 289, and 353 mμ (ϵ 43,200, 35,700, 40,100, and 37,600, respectively).

Anal. Calcd for $C_{28}H_{20}O_2$: C, 86.66; H, 5.16. Found: C, 86.32; H, 5.52.

Photochemical Isomerization of VI. A solution of VI (10 mg) in chloroform (5 ml) was irradiated with diffuse English sunlight for 30 min. The red crystals were filtered and recrystallized from dimethyl sulfoxide to give the **all-*trans* isomer VIII** as scarlet needles which sublimed at 330–332° slight decomposition without melting. VIII sublimed readily at 180° (0.1 mm) without decomposition. The mass spectrum exhibited a very prominent base peak at *m/e* 388.1465 due to the molecular ion. The infrared spectrum (KBr disk) showed prominent bands at 1625, 1012, 990, 965, 953, and 938 cm^{-1}. The ultraviolet spectrum ($CHCl_3$) showed maxima at 303 (sh), 314, 358, and 375 mμ (ϵ 45,100 52,300, 60,500, and 84,100, respectively). The nmr spectrum (60 Mcps P.D.P. 8/S CAT, dimethyl sulfoxide) showed two multiplets at τ 2.40–2.65 (4 H) and 2.70–2.95 (4 H) due to the benzenoid protons, an AB quartet centered at 3.09, 3.49 (8 H, J = 12 cps) due to the olefinic protons, and singlet at 3.72 (4 H) due to the furan ring protons.

Anal. Calcd for $C_{28}H_{20}O_2$: C, 86.66; H, 5.16. Found: C, 86.80; H, 5.48.

***cis,cis*-α,α′-Diphenyl-β,β′-(2,5-furan)diacrylic Acid (XIII).** 2,5-Furandialdehyde (2.48 g), phenylacetic acid (4.08 g), acetic anhydride (20 ml), and triethylamine (2 ml) were boiled under reflux for 30 min. Water (20 ml) was added and the reaction mixture boiled for a further 5 min. Concentrated hydrochloric acid (5 ml) was added and the reaction mixture cooled in an ice–salt bath. The crude acid was filtered, washed with water and dried. Recrystallization from glacial acetic acid (charcoal) gave the pure acid XIII (5 g, 69%) as fine yellow needles, mp 266–268°. The nmr spectrum (60 Mcps, CD_3COCD_3) exhibited a singlet at τ 2.45 (2 H, vinylic protons), a series of peaks at 2.50–2.78 (10 H, benzenoid protons), and singlet at 4.25 (2 H, furan protons).

Anal. Calcd for $C_{22}H_{16}O_5$: C, 73.31; H, 4.48. Found: C, 73.18; H, 4.28.

2,5-Di(β-phenylvinyl)furan. The carboxylic acid XIII (1.0 g), copper chromite (0.5 g), and quinoline (10 ml) were heated in an oil bath so that the internal reaction temperature was maintained at 200–210° for 1.25 hr. The reaction mixture was cooled, poured into excess 3 *N* hydrochloric acid, and extracted several times with ether. The combined ethereal extracts were washed several times with 3 *N* hydrochloric acid, with water, and finally with saturated sodium bicarbonate solution, dried (Na_2SO_4), and concentrated. The residue was adsorbed on to a column of silicic acid (Mallinckrodt, 100 mesh, column 25 × 2.75 cm) and eluted with 2% ether–pentane. Fractions (50-ml) were collected. Fractions 10–12 were concentrated, and the residue was adsorbed onto a silica gel plate (20 × 20 × 0.1 cm, Merck $HF_{254+366}$). The plate was eluted with pentane, and two distinct yellow bands developed. These bands were removed in turn and extracted into ether, and the ether extracts concentrated.

The initial yellow band yielded ***cis,cis*-2,5-di(β-phenylvinyl)furan** (XII, 133 mg, 17.6%) as a pale yellow oil. The mass spectrum showed a base peak at *m/e* 272 due to the molecular ion. The infrared spectrum (CCl_4) exhibited prominent bands at 3060, 3020, 1630, 1600, and 1028 cm^{-1}. The nmr spectrum (60 Mcps, CCl_4) exhibited a multiplet at τ 2.57–2.85 (10 H, benzenoid protons), a double doublet centered at 3.64 (4 H, J = 3 cps, olefinic protons) and a singlet at 3.88 (2 H, furan protons). The electronic spectrum (ethanol) showed maxima at 233, 273, 280 (sh), and 355 mμ (ϵ 14,300, 14,300, 13,500, and 21,700, respectively).

Anal. Calcd for $C_{20}H_{16}O$: C, 88.20; H, 5.92. Found: C, 88.43; H, 5.78.

The slower moving yellow band yielded ***cis,trans*-2,5-di(β-phenylvinyl)furan** (XIII, 106 mg, 14%) which crystallized from pentane as slender yellow needles, mp 142–144°. The mass spectrum showed a base peak at *m/e* 272 due to the molecular ion. The infrared spectrum (CCl_4) showed prominent bands at 3060, 3020, 1630, 1600, 1022, 982, and 960 cm^{-1}. The nmr spectrum (60 Mcps, CCl_4) exhibited a complex band at τ 2.42–2.70 (10 H, benzenoid protons), an AB quartet centered at 3.0, 3.28 (2 H, J = 6 cps, *trans*-olefinic protons), a triplet centered at 3.60 (2 H, J = 3 cps, *cis*-olefinic protons), and singlets at 3.23, 3.79 (each 1 H, furan protons). The ultraviolet spectrum (ethanol) showed maxima at 235, 243, 251, 274, 282, 366 (sh), 382, and 396 (sh) mμ (ϵ 15,700, 14,600, 13,100, 22,700, 21,800, 34,900, 52,400, and 33,600, respectively).

Anal. Calcd for $C_{20}H_{16}O$: C, 88.20; H, 5.92. Found: C, 87.99; H, 6.07.