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9. Complete details on the X-ray crystal structure of **2** (Li^+/ 15-Crown-5) will be presented in the full paper describing the reactivity differences between the lateral and diagonal isomer of $CpRe(CO)_2Br_2$.
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A Facile Synthetic Route to 2-[p-(2',2',3',3'-Tetracyanocyclopropyl)phenoxyl]ethanol

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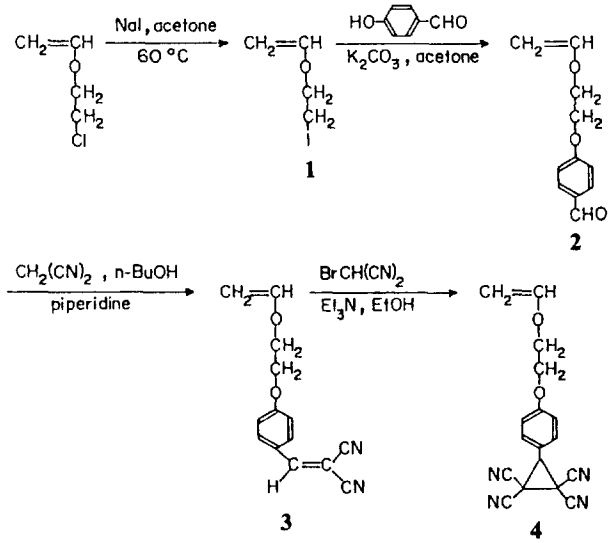
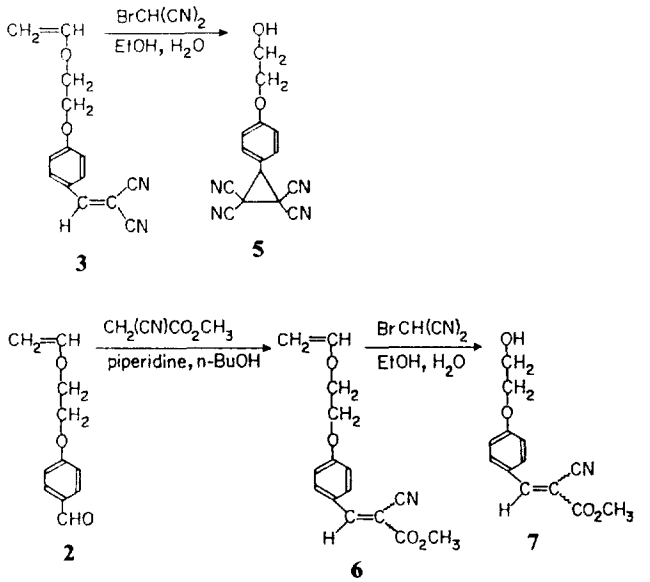
1,1,2,2-Tetracyanocyclopropane can be obtained easily by reaction of aqueous formaldehyde and malononitrile[1], tetracyanoethylene with diazomethane[2] or tetracyanoethylene with bromoketene acetals.[3] A large number of substituted 1,1,2,2-tetracyanocyclopropanes are prepared by the Wideqvist reaction,[4,5] in which a carbonyl compound react with excess bromomalononitrile. A similar cyclopropanation procedure was reported by Hart.[6,7] 1,1,2-Tricyanocyclopropanes can be prepared from ylidenecyanoacetate and bromomalonoitrile.[8] Recently, we extended the Wideqvist reaction to prepare substituted 1,2-dicyanocyclopropanes.[9]

In the course of our study of tetracyanocyclopropane,[10,11] we found that benzylidenemalononitrile or benzylidencyanoacetate containing vinyl ether moiety shows an abnormal cyclopropanation. In this work we investigated the cyclopropanation behaviors of p-(2-vinyloxyethoxy)benzylidenemalononitrile **3** and methyl (2-vinyloxyethoxy)benzylidenecyanoacetate **6**. We now report the results of the initial phase of the work.

2-Iodoethyl vinyl ether **1** was prepared by the well known halogen exchange reaction[12] from 2-chloroethyl vinyl ether and sodium iodide. Compound **1** was reacted with 4-hydroxy-



Scheme 1.



Scheme 2.

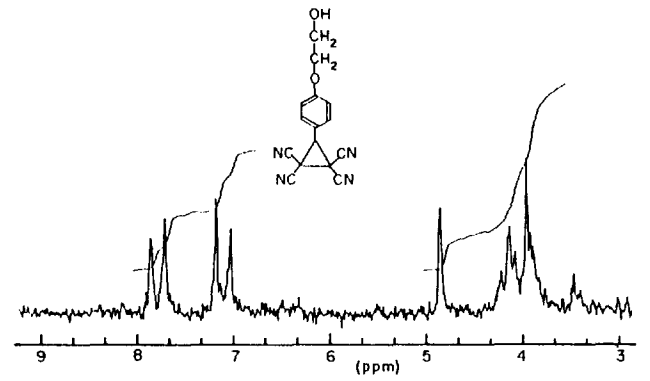


Figure 1. ^{1}H NMR spectrum of 2-[p-(2',2',3',3'-tetracyanocyclopropyl)phenoxy] ethanol **5** taken in acetone-d_6 at room temperature.

benzaldehyde to give p-(2-vinyloxyethoxy)benzaldehyde **2**.[13] Compound **2** was condensed with malononitrile or methyl cyanoacetate with a small amount of piperidine to yield p-(2-vinyloxyethoxy)benzylidenemalononitrile **3** and methyl p-(2-vinyloxyethoxy)benzylidenecyanoacetate **6**, respectively. Under aqueous ethanol solution without base such as triethylamine, which is a normal Wideqvist reaction condition, 2-[p-(2',2',3',3'-tetracyanocyclopropyl)phenoxy]ethanol **5** and acetaldehyde were obtained instead of 2-[p-(2',2',3',3'-tetracyanocyclopropyl)phenoxy]ethyl vinyl ether **4** by the reaction of **3** with bromomalonoitrile.

These experimental data indicate that the vinyl ether moiety proceeds hydrolysis by HBr released from bromomalononitrile during the cyclopropanation. However, in the presence of triethylamine, hydrolysis by HBr was inhibited and compound **4** was obtained in moderate yields from **3** and bromomalononitrile. This is because that triethylamine base captured the HBr acid to form triethylammonium bromide, which was isolated in crystals. The reactivity of methyl p-(2-vinyloxyethoxy)benzylidenecyanoacetate **6** toward bromomalononitrile was rather low, and only hydrolysis was proceeded to yield methyl p-(2-hydroxyethoxy)benzylidencyanoacetate **7** and acetaldehyde without cyclopropanation. In the presence of triethylamine base, compound **6** did not react with bromomalonoitrile. Similar experimental results were obtained when used pyridine as a base instead of triethylamine. The chemical structure of the compounds was determined by ^{1}H NMR (Figure 1), IR spectra (Figure 2), and elemental analyses. The striking structural feature of the compound **5** and **7** revealed by spectra is that these compounds do not have any unsaturation. The ^{1}H NMR spectra of compound **5** and **7** do not show any resonance peaks at 6.30-6.75 ppm attibutable to vinyl protons, as shown in Figure 1. The same compound samples showed strong absorption bands at 3385 cm^{-1} and 3495 cm^{-1} in their IR spectra (Figure 2) indicating the presence of alcoholic hydroxy groups. With those spectral data we confirmed the chemical structure of the compounds.

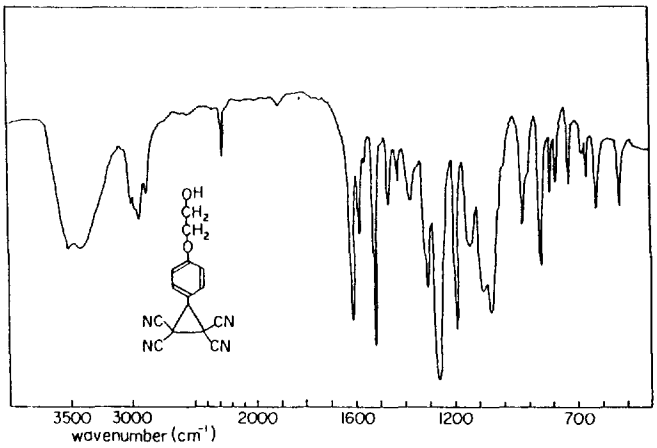


Figure 2. IR spectrum of 2-[p-(2',2',3',3'-tetracyanocyclopropyl) phenoxy] ethanol **5**.

In conclusion, p-(2-vinyloxyethoxy)benzylidenemalononitrile **3** and methyl p-(2-vinyloxyethoxy)benzylidenecyanoacetate **6** were prepared by the condensation of p-(2-vinyloxyethoxy) benzaldehyde **2** with malonoitrile or methyl cyanoacetate, respectively. We found that under neutral conditin without amine base, vinyl ether **3** proceeded an abnormal cyclopropanation and hydrolysis with bromomalononitrile to give 2-[p-(2',2',3',3'-tetracyanocyclopropyl)phenoxy]ethanol **5** in moderate yields instead of 2-[p-2',2',3',3'-tetracyanocyclopropyl) phenoxyl]ethyl vinyl ether **4**. These experimental evidence indicated that hydrolysis reaction proceeds by HBr relased during the cyclopropanation. In the presence of amine base tetracyanocyclopropane-substituted ethyl vinyl ether **4** was obtained from dicyano-substituted compound **3** and bromomalononitrile, because amine base neutralize HBr and inhibits hydrolysis of vinyl ether. Monocyano-sustituted vinyl ether **6** did not cyclopropanize with bromomalononitrile, but only to yield substituted ethanol methyl p-(2-hydroxyethoxy) benzylidenecyanoacetate **7** in the presence of amine base. All the ethyl vinyl ether derivatives (**1, 2, 3, 4, 6**) were very sensitive to acid or electrophile, polymerizing readily.

Materials. The reagent grade chemicals were purified by distillation of recrystallization before use. Bromomalonoitrile was prepared according to a literature procedure[14] and recrystallized twice from chloroform.

Measurements. IR spectra were taken on a Hitachi Model 260-30 infrared spectrophotometer. ^{1}H NMR spectra were obtained on a Varian EM 360L NMR spectrometer (60 MHz). Elemetal analyses were performed using a Perkin-Elmer 2400 CHN elemental analyzer. Melting points were measured in Buchi 530 melting point apparatus and are corrected.

2-Iodoethyl Vinyl Ether 1. Anhydrous sodium iodide (45 g, 0.30 mol) was dissolved in 320 mL of dry acetone contained in a 500 mL round-bottom flask fitted with a reflux condenser protected by a calcium chloride tube, and heated on a heating mantle for 1 h with stirring. 2-Chloroethyl vinyl ether (25.0 g, 0.23 mol) was added to the mixture, and refluxed for an additional 25 h with stirring to complete the reaction. The resulting solution was cooled to room temperature and filtered with suction. The sodium chloride on the filter was washed with 55 mL of acetone and the filtrate was concentrated by distillation of about 260 mL of the solvent. The residue was poured into 260 mL of water contained in a separate funnel, which was shaken. The lower layer was washed successively with 55 mL of 10% sodium bisulfite solution, 55 mL of 5% sodium bicarbonate, and 30 mL of water. It was dried with anhydrous magnesium sulfate (1.8 g) and fractionated under reduced pressure to give 32.8 g (72% yield) of **1**; Bp: 50-52 ℃ (20 mmHg). ^{1}H NMR ($CDCl_3$) δ 6.22-6.57 (q, 1H), 3.50-4.37 (m, 4H), 2.82-3.47 (t, 2H). IR (neat) 3105, 3034, 2960, 2922, 2845 (C-H), 1635, 1612 (C=C) cm^{-1}.

p-(2-Vinyloxyethoxy)benzaldehyde 2. 4-Hydroxybenzaldehyde (12.2 g, 0.10 mol), anhydrous potassium carbonate (18.0 g, 0.13 mol), and 2-iodoethyl vinyl ether (25.7 g, 0.13 mol) were dissolved in 180 mL of dry acetone under nitrogen. The mixture was refluxed in an oil bath kept at 60 ℃ for 72 h under nitrogen. The resulting solution was cooled to room temperature, filtered, and the inorganic salts were washed with 50 mL of acetone. Rotary evaporation of acetone gave crude product, which on vacuum distillation yielded 16.3 g (85% yield) of pure product 2: Bp: 72-74 ℃ (0.2 mmHg). Compound **2** was crystallized in the refrigerator (6 ℃). ^{1}H NMR (acetone-d_6) δ 9.89 (1H, s), 7.63-7.99 (2H, m), 6.87-7.28 (2H, m), 6.32-6.73 (1H, q), 4.18-4.48 (2H, m), 3.70-4.17 (4H, m). IR (neat) 3119, 3068, 2940, 2878, 2833 (C-H), 1694, 1603, 1579 (C=C) cm^{-1}.

p-(2-Vinyloxyethoxy)benzylidenemalononitrile 3. Piperidine (0.13 g, 1.5 mmol) was added to a solution of p-(2-vinyloxyethoxy)benzaldehyde **2** (5.38 g, 28 mmol) and malononitrile (1.98 g, 30 mmol) in 50 mL of n-butanol with stirring at 0 ℃ under nitrogen. After stirring for 1 h at 0 ℃, the product was filtered and washed with successively with cold n-butanol (80 mL), water (30 mL), and cold n-butanol (20 mL). The obtained pale yellow product was recrystallized from n-butanol to give 5.38 g (80% yield) of **3**: mp. 80-82 ℃. ^{1}H NMR (acetone-d_6) δ 7.75-8.13 (3H, m), 6.83-7.28 (2H, m), 6.30-6.73 (1H, q), 4.20-4.51 (2H, m), 3.76-4.21 (4H, m). IR (KBr) 3105, 3067, 2938, 2869 (C-H), 2230 (CN), 1628, 1603, 1579 (C=C) cm^{-1}. Anal. Calcd for $C_{14}H_{12}N_2O_2$: C, 69.99; H, 5.03; N, 11.66. Found: C, 69.90; H, 5.08; N, 11.58.

2-[p-(2′,2′,3′,3′-Tetracyanocyclopropyl)phenoxy] ethyl Vinyl Ehter 4. Bromomalononitrile (1.30 g, 9 mmol) was added to a solution of p-(2-vinyloxyethoxy)benzylidenemalononitrile **3** (1.44 g, 6 mmol) and triethylamine (0.91 g, 9 mmol) in 20 mL of absolute ethanol with stirring at 0 ℃ under nitrogen. The reaction mixture was stirred for 5 min at 0 ℃ and 12 h at room temperature. The resulting solution was cooled in an ice bath for 2 h. After filtration of the product, it was rinsed with 100 mL of water and 20 mL of cold ethanol. The obtained white product was recrystallized from ethanol-acetone (90/10. vol/vol) mixture to give 1.05 g (58% yield) of **4**: mp. 124-126 ℃ (dec). ^{1}H NMR (acetone-d_6) δ 7.52-7.92 (2H, m), 6.81-7.25 (2H, m), 6.27-6.75 (1H, q), 4.83 (1H, s), 4.17-4.42 (2H, m), 3.76-4.16 (4H, m). IR (KBr) 3062, 2988, 2910, 2865 (C-H), 2260 (CN), 1613, 1579 (C=C) cm^{-1}. Anal. Calcd for $C_{17}H_{12}N_4O_2$: C, 67.10; H, 3.97; N 18.41. Found: C, 67.18; H, 3.92; N, 18.35.

2-[p-(2′,2′,3′,3′-Tetracyanocyclopropyl)phenoxy] ethanol 5. Bromomalononitrile (1.09 g, 7.5 mmol) was added to a solution of **3** (1.20 g, 5 mmol) in 20 mL of 85% aqueous ethanol with stirring at room temperature. The reaction mixture was stirred for 3 h at 30 ℃ and 16h at room temperature. The resuting solution was cooled in an ice bath for 2 h. After filtration of the produt, it was rinsed with 60 mL of water and 20 mL of cold 85% aqueous ethanol. The obtained white product was recrystallized from acetone to give 0.78 g (56% yield) of **5**; mp. 160-162 ℃ (dec). ^{1}H NMR (acetone-d_6) δ 7.57-7.93 (2H, m), 6.90-7.28 (2H, m), 4.83 (1H, s), 3.77-4.37 (5H, m). IR (KBr) 3385 (O-H), 3035, 2998, 2928, 2865 (C-H), 2255 (CN), 1609, 1578 (C=C) cm^{-1}. Anal. Calcd for $C_{15}H_{10}N_4O_2$: C, 64.74; H, 3.62; N, 20.13. Found: C, 64.65; H, 3.58; N, 20.22.

Methyl p-(2-Vinyloxyethoxy)benzylidenecyanoacetate 6. Piperidine (0.26 g, 3.0 mmol) was added to a solution of p-(2-vinyloxyethoxy)benzaldehyde **2** (5.77 g, 30 mmol) and methyl cyanoacetate (2.97 g, 30 mmol) in 45 mL of n-butanol with stirring at 0 ℃ under nitrogen. The resulting solution was stirred for 1 h at 0 ℃ and 10 h at room temperature. After cooling in an ice bath for 2 h, the product was filtered and washed with sucessively with cold n-butanol (30 mL), water (50 mL), and cold n-butanol (20 mL). The obtained pale yellow product was recrystallized from n-butanol to give 5.90 g (72% yield) of **6**; mp. 95-97 ℃. ^{1}H NMR (acetone-d_6) δ 7.84-8.25 (3H, m), 6.93-7.29 (2H, m), 6.32-6.75 (1H, q), 4.25-4.50 (2H, m), 3.87-4.24 (4H, m), 3.88 (3H, s). IR (KBr) 3103, 3016, 2957, 2930 (C-H), 2220 (CN), 1726 (C=O), 1618, 1588 (C=C) cm^{-1}. Anal. Calcd for $C_{15}H_{15}NO_4$: C, 65.92; H, 5.53; N, 5.13. Found: C, 65.85; H, 5.49; N, 5.18.

Methyl p-(2-Hydroxyethoxy)benzylidenecyanoacetate 7. Bromomalononitrile (1.45 g, 10 mmol) was added to a solution of p-(2-vinyloxyethoxy)benzylidenecyanoacetate **6** (1.37 g, 5 mmol) in 10 mL of 85% aqueous ethanol with stirring at room temperature. The reaction mixture was stirred for 3 h at 30 ℃ and 15 h at room temperature. The resuting solution was cooled in the refrizerator (8 ℃) for 10h. After filtration of the product, it was rinsed with 80 mL of water and 20 mL of cold 85% aqueous ethanol. The obtained white product was recrystallized from acetone to give 0.84 g (68% yield) of **7**; mp. 131-133 ℃ (dec). ^{1}H NMR (acetone-d_6) δ 7.87-8.28 (3H, m), 6.93-7.29 (2H, d), 4.03-4.36 (2H, m), 3.53-4.02 (6H, m). IR (KBr) 3495 (O-H), 3025, 3002, 2950, 2862, (C-H), 2230 (CN), 1722 (C=O), 1585, 1558 (C=C) cm^{-1}. Anal. Calcd for $C_{13}H_{13}NO_4$: C, 63.15; H, 5.30; N, 5.67. Found: C, 63.25; H, 5.25; N, 5.61.

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Synthesis of 2-Substituted-4-chloromethylfurans Using 2-(Chloromethyl)-3-(trimethylsilyl)propene

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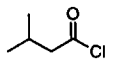
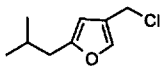
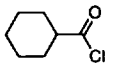
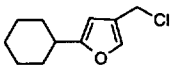
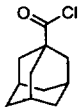
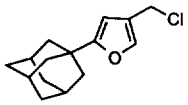
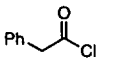
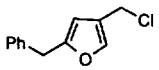
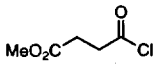
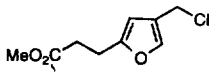
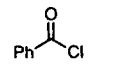
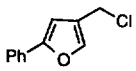
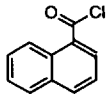
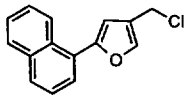
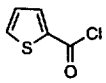
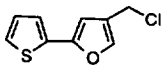
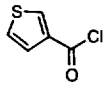
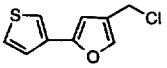
The 3-furylmethyl moiety is present in various natural products such as perillene,[1] dendrolasin,[1] amilol-A and B,[2] anonene,[3] pallescensin-1,2,3, and A,[4] pleraplysillin-1,[5] nakafuran-8 and 9.[6] Most of them were synthesized from 3-chloromethylfuran. 3-Chloromethylfuran itself has been synthesized.[1a,7] However, the reports on alkyl or aryl substituted 3-chloromethylfurans are rare.[8] Though numerous synthetic routes to furans have been developed,[9] a regioselective preparation of highly substituted furans is still demanding. We describe here a general, convenient preparation of 2-substituted-4-chloromethylfurans **5**, which are useful for the synthesis of the substituted furanosesquiterpenes.

The epoxycarbonyl compounds, good precursors of furans, have been prepared from homoallylic alcohols by epoxidation followed by oxidation.[10,11] And the homoallylic alcohols have been synthesized from the reaction of allylic chlorides with aldehydes in the presence of magnesium or zinc.[12] However, these synthetic routes have some limitations for the preparation of variously substituted epoxycarbonyl compounds. Recently, we found that allylic ketones, which were obtained from the Lewis acid-mediated reactions of allysilanes with acid chlorides,[13] could be transformed to epoxycarbonyl compounds by epoxidation with m-chloroperoxybenzoic acid (MCPBA). Following this procedure, 2-substituted-4-chloromethylfurans **5** were prepared from the allysilane, 2-(chloromethyl)-3-(trimethylsilyl)propene (**1**)[14] as shown in the Scheme.

Table 1. Synthesis of 2-Substituted-4-chloromethylfurans **5**[a]

Entry	Acid Chloride **2**	Furan **5**	Overall Yield,[b] %
a			57
b			49
c			44
d			47
e			35
f			43
g			42
h			46
i			53

[a] The three step reactions were carried out consecutively without isolation of the intermediate products (See text). [b] Isolated yields (not optimized).

Reaction of allysilane **1** with isovaleryl chloride (**2a**) in the presence of one equiv of $TiCl_4$ at -78 ℃ in CH_2Cl_2 led to the allylic ketone **3a** in 92% yield after chromatography (SiO_2, hexane : ether = 8 : 1). The possible formation of conjugated enones coming from an acidic isomerization of **3** was not observed under our experimental conditions. Epoxidation of **3a** with MCPBA (2 equiv) in CH_2Cl_2 at 0 ℃ ~ room temperature gave the epoxide **4a** in 85% yield after purification (SiO_2, CH_2Cl_2). When a benzene solution of **4a** with a catalytic amount of p-toluenesulfonic acid was refluxed for 1 h, 2-isobutyl-4-chloromethylfuran **5a** was produced in 66% yield after purification by molecular distillation. The furan products **5**, especially aryl substituted ones, were lost significantly during chromatography on silica gel and alumina.

The three step procedures, the $TiCl_4$-promoted reaction of allylsilane **1** with an acid chloride, epoxidation with MCPBA, and cyclization process could be performed successively