One-Pot Preparation of α-Cyanovinyl Ethers (2-Alkoxy-2-alkenenitriles) from Vinyl Ethers: Elaboration to 3-Alkoxy-2-oxo-3-alkenenitriles and Aluminium Chloride-Catalyzed Cycloadditions to Cyclopentadiene

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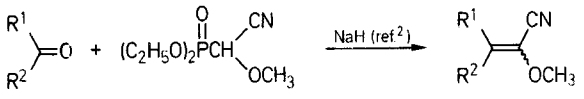
A variety of α-cyanovinyl ethers have been prepared by 3 different routes and elaborated to 3-alkoxy-2-oxo-3-alkenenitriles. The latter, in the presence of cyclopentadiene and aluminium chloride, give novel bridged 7-membered cycloadducts with loss of hydrogen cyanide.

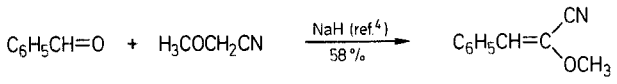
2-Alkoxy-2-alkenenitriles **2** are versatile synthetic intermediates which have served, for example, as donor-acceptor substituted olefins for free radical reactions[1], as intermediates in a one-carbon homologation of carbonyl compounds[2], and also as ketene equivalents[3]. In studies directed towards the development of novel cycloaddition reagents, we have investigated the preparation of various α-cyanovinyl ethers. The compounds were also converted into 3-alkoxy-2-oxo-3-alkenenitriles (**10**), which in turn were employed in aluminium chloride-catalyzed cycloadditions to cyclopentadiene.

1. Synthesis of α-Cyanovinyl Ethers 2

Previously, several procedures have been used. The Horner-Wittig reaction with the α-alkoxy-α-cyanomethylphosphonate **1** allows methylenation of enolizable aldehydes and also of ketones, e.g. benzophenone[2]. The direct aldol-like condensation of alkoxyacetonitriles such as **3** with benzaldehyde was also been reported[4].

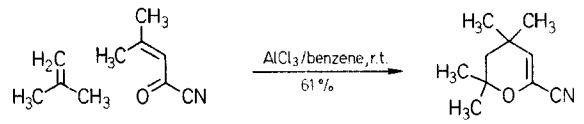


1 **2**



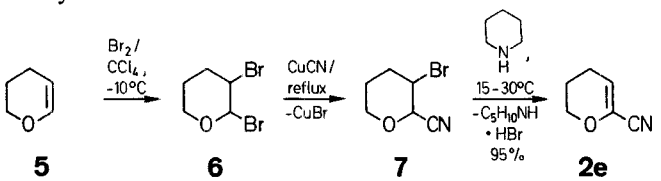
3 **2a**

However, the preparation of the Horner reagent **1** is not straightforward and cyclic representatives of **2** cannot be obtained by either process. More recently, we have prepared 6-membered α-cyanovinyl ethers, i.e. 3,4-dihydro-2*H*-pyran-6-carbonitriles by the aluminium chloride-catalyzed Diels-Alder reaction of α,β-unsaturated acyl cyanides with simple olefins[5]. For example, **2f** has now been obtained in 61% yield (Table 1).



4 **2f**

Since the parent, non-methylated heterocycle **2e** cannot be prepared in this fashion, the three-step sequence **5** → **6** → **7** → **2e** was tried. Although this approach is known in principle[6], we encountered difficulties in obtaining acceptable yields.



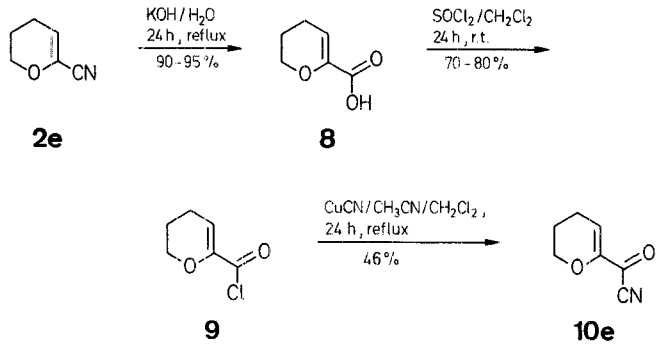
5 **6** **7** **2e**

Several modifications have proved useful for preparing α-cyanovinyl ethers. (i) Diethyl ether was replaced by the more inert and higher boiling tetrachloromethane as a solvent. This solvent appears to be advantageous for the step **6** → **7** (possibly a free radical reaction) and also during the final non-aqueous distillative work up and isolation of the product. (ii) The starting vinyl ether was brominated under as mild conditions as possible (about −10 °C and below). (iii) The piperidine induced dehydrobromination was allowed to proceed over an extended period (about 60 h) at room temperature.

In this fashion, α-cyanovinyl ethers **2** were obtained in the 60–95% range and the reaction could be scaled up from 0.1 to 0.4 molar. The three-step synthesis of α-cyanovinyl ethers from vinyl ethers as outlined here is a single flask procedure and is applicable to cyclic and also acyclic compounds.

2. 3-Alkoxy-2-oxo-3-alkenenitriles (10)

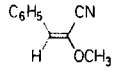
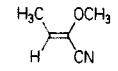
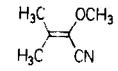
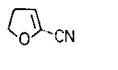
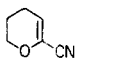
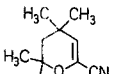
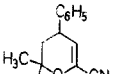
The homologation of α-cyanovinyl ethers to **10** can be exemplified by the reaction with **2e**, as follows.



2e **8** **9** **10e**

While 4,5-dihydrofuran-2-carboxylic acid could be prepared (from **2d**), the derived acid chloride is more sensitive and could not be obtained; instead, decomposition occurred. The spectroscopic data of the novel acyl cyanides **10a–f** (Table 2) agree with those of simpler derivatives.

Table 1. α-Cyanovinyl Ethers **2** by Several Procedures

Product		Method of Preparation[a]	Yield [%]	b.p. [°C]/ torr	Molecular Formula	^{1}H-N.M.R. ($CDCl_3$/TMS) δ [ppm]	I.R. (CCl_4) ν [cm^{-1}]
 E:Z = 2:1	**2a**[4]	A	58	115–128°C/10	$C_{10}H_9NO$ (159.2)	3.70 (s, 3H, OCH_3-*E*); 3.91 (s, 3H, OCH_3-*Z*); 6.16 (s, 1H, H_{olefin}-*Z*); 6.53 (s, 2H, H_{olefin}-*E*); 7.33–7.52 (m, 5H_{arom})	2205s, 1690vs, 1625s, 1285s, 1130vs
 E:Z = 45:1	**2b**	B	60[b]	–[d]	C_5H_7NO (97.1)	2.03 (d, *J* = 7 Hz, 3H, CH_3); 3.7 (s, 3H, OCH_3-*Z*); 3.75 (s, 3H, OCH_3-*E*); 6.45 (q, *J* = 7 Hz, 1H, H_{olefin}-*Z*); 6.88 (q, *J* = 7 Hz, 1H, H_{olefin}-*E*)	2230s, 1680vs, 1630vs, 1080vs
	**2c**	B	60[b]	40°C/12[d]	C_6H_9NO (111.1)	2.03 (s, 3H); 2.18 (s, 3H); 3.73 (s, 3H, OCH_3)	2210s, 1685vs, 1605vs, 1280vs
	**2d**[7]	B	63	64–65°C/14	C_5H_5NO (95.1)	2.82 (dt, *J* = 3 Hz, *J* = 10 Hz, 2H, CH_2); 4.47 (t, *J* = 10 Hz, 2H, OCH_2); 5.89 (t, *J* = 3 Hz, 1H, H_{olefin})	3120m, 2980m, 2245s, 1630s, 1175vs
	**2e**[8]	B	95	95–96°C/20	C_6H_7NO (109.1)	1.77–2.1 (m, 2H, CH_2); 2.31–2.56 (m, 2H, CH_2); 4.17 (t, *J* = 5 Hz, OCH_2); 5.67 (t, *J* = 5 Hz, H_{olefin})	2220s, 1640vs, 1250vs, 1090vs
	**2f**[5]	C[c]	61	–[d]	$C_{10}H_{15}NO$ (165.2)	1.11 (s, 6H); 1.31 (s, 6H); 1.66 (s, 2H); 5.48 (s, 1H)	2950vs, 2220s, 1628vs, 1110vs
	**2g**	C[c]	93	120°C/0.1[d]	$C_{14}H_{15}NO$ (213.1)	1.37 (s, 6H, $2CH_3$); 1.86 (m, 2H); 3.52 (m, 1H, CH); 5.66 (m, 1H, H_{olefin}); 7.26 (m, 5H_{arom})	2980s, 2235s, 1635vs, 1600m, 1105vs, 1025s

[a] A: Aldol-like condensation[4]; B: Single-flask reaction starting from vinyl ether; C: aluminium chloride-catalyzed cycloaddition[5].
[b] Not optimized.
[c] Reaction time was 3d at 0°C in benzene.
[d] Kugelrohr distillation.

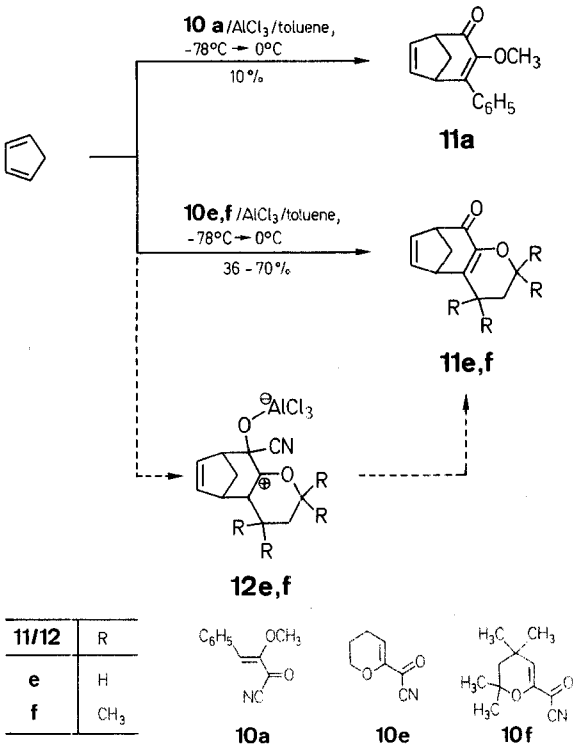
Table 2. 3-Alkoxy-2-oxo-3-alkenenitriles **10** (R—CO—CN) from **2** (R—CN)

Product	Yield[a] [%]	b.p. [°C]/ torr[c]	Molecular Formula[b]	^{1}H-N.M.R. ($CDCl_3$/TMS) δ [ppm]	I.R. (CCl_4) ν [cm^{-1}]
10a[4]	64	100°/0.1	$C_{11}H_9NO_2$ (187.2)	3.88 (s, 3H, OCH_3); 7.34 (s, 1H, H_{olefin}); 7.48–7.93 (m, 5H, H_{arom})	3030w, 2950m, 2210s, 1700s, 1625s, 1242vs, 1150s, 1140s
10b	36	80–90°/5	$C_6H_7NO_2$ (125.1)	2.03 (d, *J* = 7 Hz, 1H); 3.7 (s, 3H, OCH_3-*Z*); 3.75 (s, 3H, OCH_3-*E*); 6.45 (q, *J* = 7 Hz, 1H_{olefin}-*Z*); 6.88 (q, *J* = 7 Hz, 1H_{olefin}-*E*)	2950s, 2230s, 1680vs, 1630vs, 1250s, 1080vs
10c	60	80–90°/5	$C_7H_9NO_2$ (139.1)	2.03 (s, 3H); 2.18 (s, 3H); 3.73 (s, 3H, OCH_3)	3010m, 2940s, 2220s, 1685vs, 1605vs, 1280vs, 1210vs, 1020s
10e	46	50°/0.1	$C_7H_7NO_2$ (137.1)	1.77–2.11 (m, 2H, CH_2); 2.31–2.56 (m, 2H, CH_2); 4.17 (t, *J* = 5 Hz, 2H, OCH_2); 6.61 (t, *J* = 5 Hz, 1H, H_{olefin})	3065w, 2940s, 2210s, 1680vs, 1620vs, 1250vs, 1090vs, 1070vs, 1000s
10f	46	110°/0.1	$C_{11}H_{15}NO_2$ (193.3)	1.24 (s, 6H); 1.36 (s, 6H); 1.74 (s, 2H); 6.42 (s, 1H)	2990m, 2215m, 1690vs, 1620s, 1210m, 1060s

[a] Relative to **2**.
[b] Satisfactory high resolution mass spectra obtained.
[c] Kugelrohr distillation.

3. Cycloadditions

Previously, simple α,β-unsaturated acyl cyanides and π systems have been found to react in a fairly predictable fashion. Thus, in the presence of aluminium chloride, cyclopentadiene and 3,3-dimethylacryloyl cyanide give conventional norbornene derived Diels-Alder adducts and also Diels-Alder adducts of inverse electron-demand, i.e. dihydropyrans[5] (cf. also preparation of **2f, g**, Table 1). Attempted reaction of **10b** and **10c** with cyclopentadiene in the presence of aluminium chloride gave polymers and ill-defined products. Interestingly, **10a** yielded **11a**, while the cyclic precursor **10f** reacted more readily, giving **11f**. The less hindered **10e** and cyclopentadiene combined to **11e** in respectable 70% yield. A plausible precursor of the tricycles **11e,f** is oxonium ion **12e,f**: apparently, complexes of the reagent **10** with aluminium chloride can behave as allyl cation equivalents containing a donor group, i.e. alkoxy, at the central carbon of the allyl moiety[9]. The reaction is completed by decomplexation of aluminium chloride and loss of hydrogen cyanide, which corresponds to an oxidation. The formation of **11a, e, f** from cyclopentadiene and **10a, e, f** represents a new and unexpected reaction of α,β-unsaturated acyl cyanides.



11/12	R
e	H
f	CH_3

6-Cyano-3,4-dihydro-2*H*-pyran (2e) (Method B):
A 2 l three necked flask equipped with a low temperature thermometer, drying tube and dropping funnel is charged with a solution of 3,4-dihydro-2*H*-pyran (71.5 g, 0.85 mol) in carbon tetrachloride (425 ml), which is cooled to −10 °C (methanol/Dry Ice). Bromine (137.3 g, 44 ml, 0.86 mol) in carbon tetrachloride (15 ml) is added dropwise at −6 to −12 °C (internal temperature) during 1 h. The mixture is allowed to reach room temperature, and stirred for a further 1.5 h. The resulting brown solution is treated with copper(I) cyanide (80.6 g, 0.90 mol) and refluxed for 16 h under vigorous stirring, while the brown-green suspension changes colour to whitish brown. The solid is filtered off without delay, the filtrate is treated with piperidine (74 g, 86 ml, 0.87 mol) and stirred well. Within 30 min a vigorous reaction sets in and the temperature is held between 15 and 30 °C [if the reaction becomes too vigorous, piperidine hydrobromide precipitates and may block the stirrer; in this case addition of further carbon tetrachloride (200 ml) is necessary]. After 60 h at room temperature, the precipitated piperidine hydrobromide is filtered off, the filtrate is washed with water (4 × 100 ml), and dried with sodium sulfate. After removal of carbon tetrachloride, distillation gives the product **2e**; yield: 87.66 g (95%); b.p. 52.5 °C/0.15 torr; purity: > 99.99% by G.L.C.; see Table **1**.

α,β-Unsaturated Carboxylic Acids 8:
A mixture of unsaturated nitrile **2** (0.056 mol) is refluxed with potassium hydroxide (6.47 g, 0.12 mol) in water (37 ml) for 24 h. The reaction mixture is acidified to pH 1 with dilute hydrochloric acid, saturated with sodium chloride and extracted with ether (6 × 60 ml). The combined ether phase is dried with sodium sulfate, the solvent is removed, and the crude product is distilled (Kugelrohr), giving the carboxylic acids **8**; yield: 90–95%.

α,β-Unsaturated Carboxyl Chlorides 9:
The carboxylic acid **8** (0.027 mol) in absolute dichloromethane (20 ml) is added dropwise to a solution of thionyl chloride (0.042 mol) in absolute dichloromethane (10 ml) at room temperature. The resulting solution is stirred for 24 h and the solvent together with the excess of thionyl chloride is removed under reduced pressure. Distillation (Kugelrohr) of the remaining oil affords the acid chloride **9**; yield: 70–80%.

α,β-Unsaturated Ketonitriles 10:
A flamed out 50 ml two-necked flask is charged with copper(I) cyanide (2.7 g, 30 mmol), absolute acetonitrile (20 ml) and dichloro-

Table 3. Cycloadducts from Aluminium Chloride-Catalyzed Reactions of **10** and Cyclopentadiene

Cycloadduct 11	Yield [%]	Molecular Formula[a]	^{1}H-N.M.R. ($CDCl_3$/TMS) δ [ppm]	I.R. (CCl_4) ν [cm^{-1}]
11a	10	$C_{15}H_{14}O_2$ (226.1)	2.26–2.72 (m, 2H, CH_2); 3.4–3.68 (m, 2H, bridgehead H's); 3.48 (s, 3H, CH_3); 6.25 (dd, J = 3.5 Hz, J = 5.5 Hz, 1H, H_{olefin}); 6.83 (dd, J = 3.5 Hz, J = 5.5 Hz, 1H, H_{olefin}); 7.21–7.62 (m, 5H_{arom})	3070m, 2980s, 1690vs, 1145vs
11e	70	$C_{11}H_{12}O_2$ (176.1)	1.66–2.08 (m, 2H, CH_2); 2.08–2.54 (m, 4H, 2CH_2); 2.92–3.08 (m, 1H, bridgehead H); 3.32–3.47 (m, 1H, bridgehead H); 3.69–4.14 (m, 2H, OCH_2); 6.21 (dd, J = 2 Hz, J = 5.5 Hz, H_{olefin}); 6.74 (dd, 1H, J = 2 Hz, J = 5.5 Hz, H_{olefin})	3075w, 2940vs, 1690vs, 1650s, 1280s, 1165vs
11f	36[b]	$C_{15}H_{20}O_2$ (232.3)	1.13 (s, 6H, 2CH_3); 1.23 (s, 3H, CH_3); 1.26 (s, 3H, CH_3); 1.66 (s, 2H, CH_2); 2.43 (t, J = 3 Hz, 2H, CH_2); 3.38 (m, J = 3 Hz, 2H, bridgehead H); 6.18 (dd, J = 5.5 Hz, J = 3 Hz, 1H_{olefin}); 6.67 (dd, J = 5 Hz, J = 3 Hz, 1H_{olefin})	3050w, 1690vs, 1610m, 1250s, 1215s

[a] Satisfactory high resolution mass spectra obtained.
[b] Yield based on reacted **2f**; 26% of **2f** was recovered.

methane (6 ml) under an atmosphere of nitrogen. The acid chloride **9** (15 mmol) in dichloromethane (5 ml) is added slowly. The mixture is refluxed for 24 h, cooled to room temperature, and diluted with dichloromethane (10 ml). The resulting precipitate is separated and the solvent is evaporated. Distillation (Kugelrohr) of the residue affords the unsaturated acyl cyanides **10** (Table 2)[10].

Cycloadditions; 4-Oxatricyclo[7.2.1.0^{3,8}]deca-3,10-dien-2-one (11e): Nitrile **10e** (1.0 g, 7.3 mmol) and freshly distilled cyclopentadiene (0.53 g, 8.0 mmol) in toluene (25 ml) are added dropwise to a solution of aluminium chloride (0.975 g, 7.3 mmol) in toluene (5 ml) at −78 °C under nitrogen. The mixture is stirred for 12 h, while being allowed to slowly reach 0 °C, diluted with ether (30 ml), and washed with water. The organic phase is dried with magnesium sulfate and the crude product is purified by flash chromatography (ether/light petroleum = 1:1), giving **11e**; yield: 0.899 g (70%); see Table 3.

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[10] Recent synthetic routes to acyl cyanides: Hoffmann, H.M.R. Haase, K., Ismail, Z.M., Preftitsi, S., Weber, A. *Chem. Ber.* **1982**, *115*, 3880.

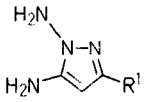
Errata and Addenda 1986

I. Ganboa, C. Palomo *Synthesis* **1986**, 52. The ^{1}H-NMR data for compounds **2d** and **2e** in the Table (p. 53) should be, respectively: 8.13 (d, $2H_{arom}$); 7.46 (d, $2H_{arom}$); 7.3 (s, $5H_{arom}$); 5.73 (m, 1 H, C–H); 5.26 (s, 2H, CH_2–$C_6H_4NO_2$); 4.9 (m, 1H, C–H); 3.7 (m, 2H, CH_2–CO–NH); 3.3 (m, 2H, S–CH_2); 2.13 (s, 3H, CH_3). 7.33 (s, $5H_{arom}$); 7.3 (s, $5H_{arom}$); 5.76 (m, 1 H, C–H); 5.2 (s, 2H, C_6H_5–CH_2); 4.9 (m, 1 H, C–H); 3.63 (s, 2 H, CH_2–CO–NH); 3.3 (m, 2H, S–CH_2); 2.13 (s, 3 H, CH_3).

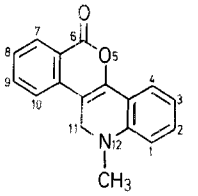
The ^{1}H-NMR data for compound **6** (p. 54) should be:
^{1}H-NMR ($CDCl_3$/TMS_{int}): δ = 8.03 (d, $2H_{arom}$); 7.43 (d, $2H_{arom}$); 5.65 (s, 1 H, CH); 5.23 (s, 2 H, CH_2); 4.5 (s, 1 H, NH); 1.53, 1.35 ppm (2 s, 6 H, 2 CH_3).

K. Tanaka, H. Yoda, K. Inoue, A. Kaji *Synthesis* **1986**, 66. The $[\alpha]_D^{25}$ value for compound **2e** in Table 1 (p. 67) should be:
$-28.2°$ (1.80).

D. R. Sliskovic, M. Siegel, Y. Lin *Synthesis* **1986**, 71. The structures for compounds **6a, b** (p. 73) should be:

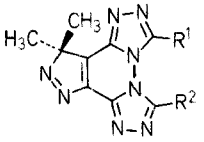


O. Meth-Cohn *Synthesis* **1986**, 76. The correct numbering for compounds **8** and **10** (p. 76) is as illustrated below for compound **10**:



10

B. Furlan, B. Stanovnik, M. Tišler *Synthesis* **1986**, 78. The double-bond arrangement of compounds **3, 6,** and **7** (pp. 78, 79) should be:

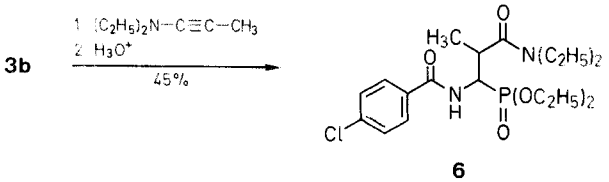


N. Petragnani, H. M. C. Ferraz, G. V. J. Silva *Synthesis* **1986**, 157. The authors wish to include the following pertinent references:
R. M. Adlington, A. G. M. Barret *Tetrahedron* **1981**, *37*, 3935.
R. M. Adlington, A. G. M. Barret *J. Chem. Soc. Perkin Trans. I* **1981**, 2848.
R. M. Adlington, A. G. M. Barret *J. Chem. Soc. Chem. Commun.* **1981**, 65.
R. M. Adlington, A. G. M. Barret *J. Chem. Soc. Chem. Commun.* **1979**, 1122.

A. J. Fatiadi *Synthesis* **1986**, 249. The heading for the first experimental procedure on p. 268 should be:
2,6-Diphenyl-4-(2,3,3-tricyanoallylidene)pyran (201)[354]:

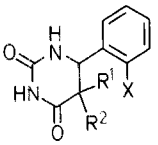
D. P. Matthews, J. P. Whitten, J. R. McCarthy *Synthesis* **1986**, 336. The headings for the first and last experimental procedures should be, respectively:
***N*^{1},*N*^{3}-Bis(2,2-dimethoxyethyl)oxaldiamidine Dihydrochloride (2):**
2-(2-Imidazolyl)-4-methoxy-4,5-dihydroimidazole (5):

T. Schrader, R. Kober, W. Steglich *Synthesis* **1986**, 372. The last equation in the formula scheme (p. 372) should be:

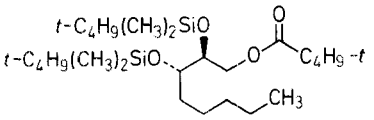


6

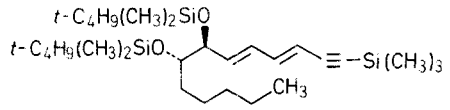
D. N. Dhar, K. S. K. Murthy *Synthesis* **1986**, 437. The heading for Table 2 (p. 440) should be:
4-Aryl-2(1*H*)-quinazolines **(13)** and 4-Aryl-1*H*-2,1,3-benzothiadiazine 2,2-Dioxides **(14)**
The names of compounds **13a** and **14a** in the experimental procedure on the same page should be corrected accordingly.
For compounds **60** and **61** (p. 445) R^3 = H, SO_2Cl.
The product in the lower, left reaction scheme on p. 446 should be:



K. C. Nicolaou, S. E. Webber *Synthesis* **1986**, 453. The structures of compounds **8** (p. 454) and **16** (p. 455) should be:



8

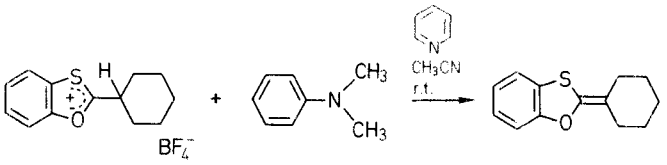


16

E. Dalcanale, M. Foà *Synthesis* **1986**, 492. In the reaction scheme, products **4** and **5** are obtained in 33 and 8%, respectively, a ratio of 80:20.

W. G. Dauben, J. M. Gerdes, G. C. Look *Synthesis* **1986**, 532. In the experimental procedure headings (p. 534), the names of compounds 3, 5, 7, and 9 should read:
(3,3-Ethylenedioxybutyl)triphenylphosphonium Bromide (3)
6-*t*-Butyldimethylsiloxy-3,7-dimethyl-1,6-octadiene (5)
5-[1,1-Bis(ethoxycarbonyl)ethyl]bicyclo[3.3.0]octan-2-one (7)
2,2-Ethylenedioxy-1,3,3-trimethylbicyclo[2.2.1]heptane (9).

S. Cadamuro, I. Degani, R. Fochi, A. Gatti, V. Regondi *Synthesis* **1986**, 544. Formula Scheme **B** should be:



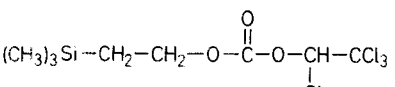
1c **2** **5**

H. M. R. Hoffmann, K. Giesel, R. Lies, Z. M. Ismail *Synthesis* **1986**, 548. The heading for the last experimental procedure (p. 551) should be:
Cycloadditions; 4-Oxatricyclo[7.2.1.0^{3,8}]dodeca-3,10-dien-2-one (11e):

Abstract 7330, *Synthesis* **1986**, 599. The structure of compound **7** should be: CH_2=C(R^6)R^7.

Abstract 7333, *Synthesis* **1986**, 600. Line 2 of the text should read: dimenthyl succinate **(1)** with lithium 2,2,6,6-tetramethylpiperidide reacts...

G. Barcelo, J. P. Senet, G. Sennyey, J. Bensoam, A. Loffet *Synthesis* **1986**, 627. The structure of compound **1k** (p. 630) should be:



D. Achet, D. Rocrelle, I. Murengezi, M. Delmas, A. Gaset *Synthesis* **1986**, 642. The last word of the title should be: **Sulfate**