1,3-Dipolar Cycloadditions of 2-Diazopropane to 1-Phenyl-bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine. The Synthesis of 11,11-Dimethyl-11 *H*-pyrazolo [3,4-*d*]bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazines, Derivatives of a Novel Heterocyclic System

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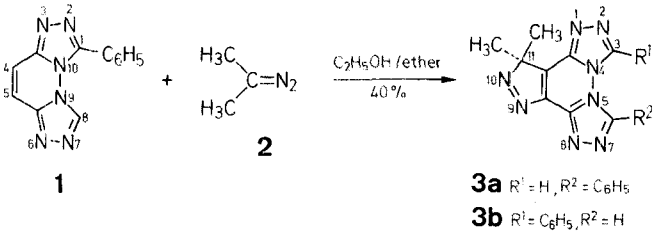
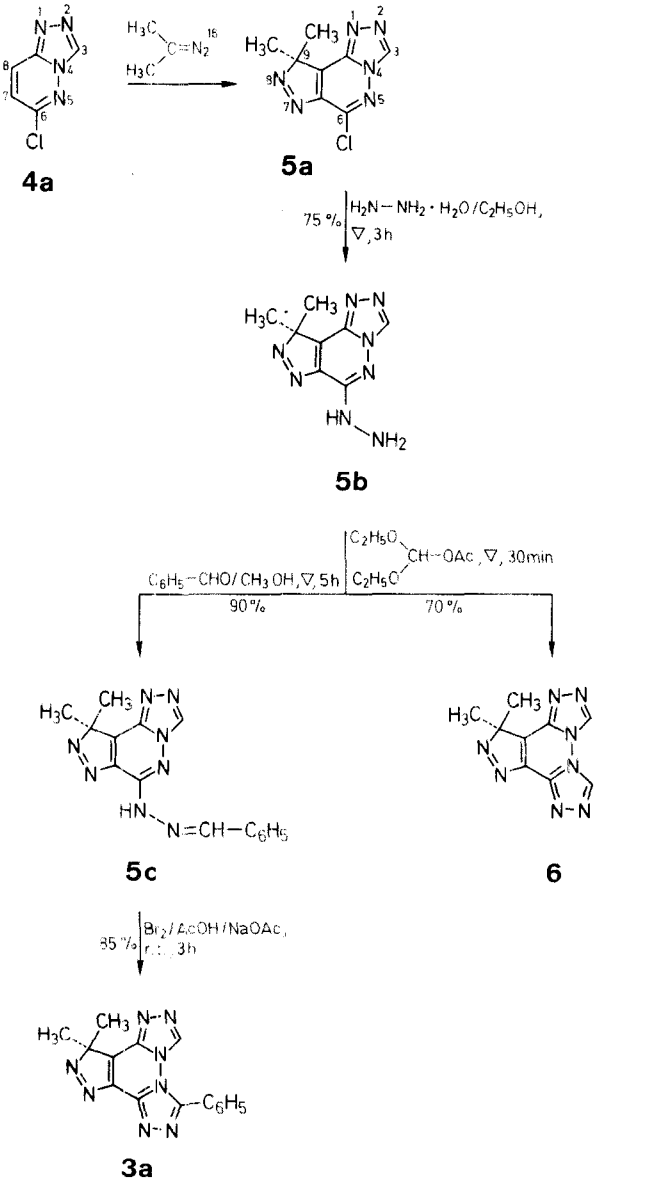
Diazopropane (**2**) undergoes [3 + 2]-cycloaddition with 1-phenyl-bis-*s*-triazole[4,3-*b*:3′,4′-*f*]pyridazine (**1**), which is a derivative of a heteroaromatic 14 π-electron system, to give a 1:1 mixture of 11,11-dimethyl-6-phenyl- (**3a**) and 11,11-dimethyl-3-phenyl-11 *H*-pyrazolo[3,4-*d*]-bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine (**3b**). These compounds represent derivatives of a novel heterocyclic system. The structures of the products were determined by independent syntheses.

1,3-Dipolar cycloadditions of diazoalkanes to systems with C=C bonds or hetero double bonds, to heterocumulenes, and to systems with triple bonds represent important methods for the synthesis of various five-membered heterocycles[1] including natural products[2,3,4]. Intermolecular and intramolecular 1,3-dipolar cycloadditions of diazoalkanes to olefins are throughly studied reactions[1,5,6,7]. On the other hand, only few examples of cycloadditions of diazoalkanes to six-membered heteroaromatic systems have been reported. The only examples described are cycloadditions to pyridine[8], to pyridazinones[9–11], to nitrofuroxanes[12], and to a substituted pyrrolo[1,2-*a*]pyrimidine[13]. In view of this fact, a systematic study of cycloadditions of diazoalkanes to azolo- and azinopyridazines with a bridgehead N-atom, and to other fully aromatic 10 π-electron systems has been undertaken. So far, the imidazo[1,2-*b*]pyrazolo[4,3-*d*]pyridazine[14], pyrazolo[4,3-*d*]- and pyrazolo[3,4-*d*]-*s*-triazolo[4,3-*b*]pyridazines, pyrazolo[4,3-*d*]- and pyrazole [3,4-*d*]tetrazolo[1,5-*b*]pyridazines[15], and pyrazolo[4,3-*d*]-pyrimido[1,2-*b*]pyridazin-4(10*H*)-ones[16] have been prepared by such cycloadditions.

In continuation of our studies in this area, we report here the cycloaddition of 2-diazopropane (**2**) to 1-phenyl-bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine (**1**), a derivative of a fully heteroaromatic 14 π-electron tricyclic system in which cycloaddition occurs across the 4,5 double bond to produce a mixture of the isomeric 11,11-dimethyl-6-phenyl- (**3a**) and 11,11-dimethyl-3-phenyl-11 *H*-pyrazolo[3,4-*d*]bis-*s*-triazolo-[4,3-*b*:3′,4′-*f*]pyridazine (**3b**) in a 1:1 molar ratio. Compounds **3a** and **3b** are derivatives of a novel heterocyclic system.



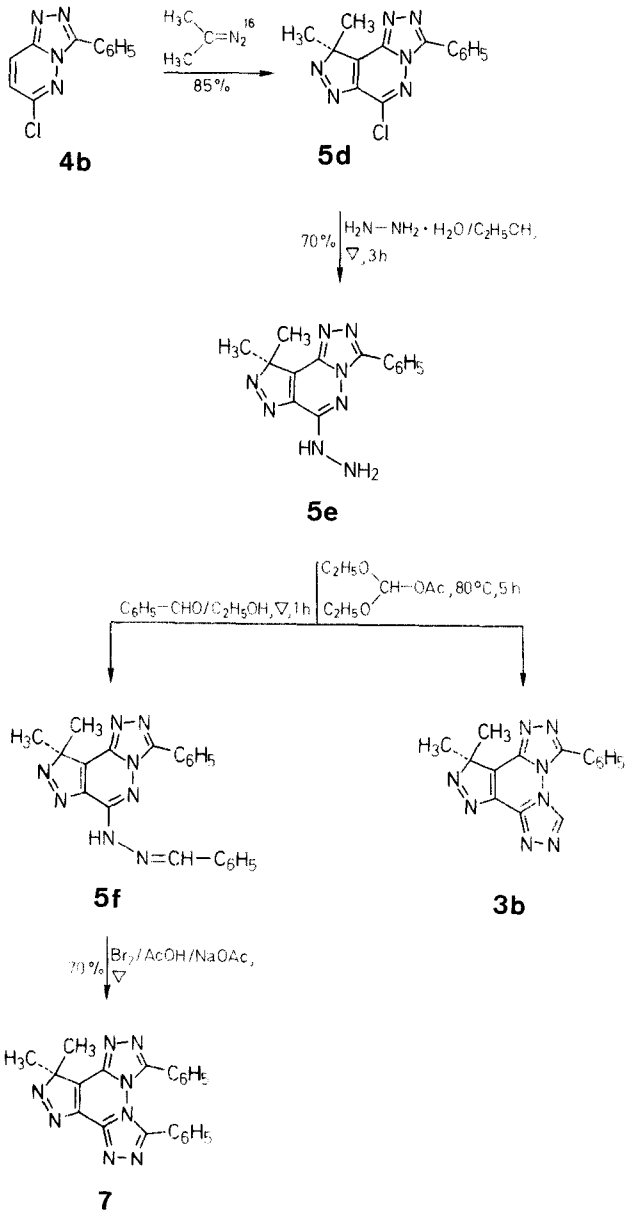
The ^{1}H-N.M.R. spectrum of the crude mixture shows two singlets of equal intensities at $\delta = 1.82$ and 1.80 ppm, each integrating for six protons, corresponding to the 11,11-dimethyl groups in isomers **3a** and **3b**, two singlets of equal intensities at $\delta = 8.50$ and 8.55 ppm, each integrating for one proton, corresponding to 3-H in isomer **3a** and 6-H in isomer **3b**; and a broad singlet at $\delta = 7.40$–7.65 ppm, integrating for 10 protons, corresponding to the 6-phenyl and 3-phenyl



groups in isomers **3a** and **3b**, respectively. Unfortunately, separation of the two isomers was not successful, even by applying flash chromatography on silica gel.

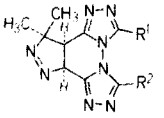
The structures of products **3a** and **3b** were determined by independent syntheses. Compound **3a** was prepared by the sequence **4a** → **5a** → **5b** → **5c** → **3a** starting from 6-chloro-*s*-triazolo[4,3-*b*]pyridazine (**4a**)[17], which was converted into 6-chloro-9,9-dimethyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**5a**) by reaction with 2-diazopropane (**2**) according to the known procedure[15]. Treatment of compound **5a** with hydrazine hydrate gave the 6-hydrazino derivative **5b** which on reaction with benzaldehyde afforded the hydrazone **5c**. Cyclodehydrogenation of **5c** with bromine in acetic acid gave the tetracyclic isomer **3a**.

The isomeric compound **3b** was prepared by the analogous sequence **4b** → **5d** → **5e** → **3b**. 6-Chloro-3-phenyl-*s*-triazolo[4,3-*b*]pyridazine[18] (**4b**) was converted into 6-chloro-9,9-dimethyl-3-phenylpyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**5d**) by reaction with 2-diazopropane[cf. 15]. Reaction of compound **5d** with hydrazine hydrate and cyclocondensation of the resultant 6-hydrazino derivative **5e** with diethoxymethyl acetate afforded the tetracyclic isomer **3b**.



Two further derivatives of the same tetracyclic system were also prepared. Thus, the cyclocondensation of hydrazino derivative **5b** with diethoxymethyl acetate gave 11,11-dimethyl-11*H*-pyrazolo[3,4-*d*]bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]-pyridazine (**6**; 70%) and condensation of hydrazine derivative **5b** with benzaldehyde and cyclodehydrogenation of the resultant hydrazone **5f** with bromine in acetic acid afforded 11,11-dimethyl-3,6-diphenyl-11*H*-pyrazolo[3,4-*d*]bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine (**7**; 70%).

The reaction of 2-diazopropane (**2**) with 1-phenyl-bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine (**1**) is assumed to proceed as a 1,3-dipolar cycloaddition of **2** to the 4,5 double bond of **1** followed by oxidation of the primary cycloadducts **8a** and **8b** by air.



8a $R^1 = H, R^2 = C_6H_5$
8b $R^1 = C_6H_5, R^2 = H$

1-Phenyl-bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine[18] (**1**), 6-chloro-9,9-dimethyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*d*]pyridazine[15] (**5a**), 6-chloro-3-phenyl-*s*-triazolo[4,3-*b*]pyridazine[18] (**4b**), and 2-diazopropane[19] (**2**) were prepared in essentially the same way as reported in the literature.

11,11-Dimethyl-6-phenyl-11*H*-pyrazolo[3,4-*d*]-bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine (3a) and 11,11-Dimethyl-3-phenyl-11*H*-pyrazolo[3,4-*d*]bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine (3b):

To a stirred solution of 1-phenyl-bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine (**1**; 236 mg, 1 mmol) in ethanol (50 ml), three freshly prepared solutions of 2-diazopropane [**2**; each time prepared from acetone hydrazone[19] (6 g) in ether (120 ml)] are consecutively added dropwise over a period of 12 h. The resultant solution is evaporated in vacuo to one third of its volume and the crystalline material isolated by suction; yield: 40%.

^{1}H-N.M.R. (DMSOd_6/TMS$_{int}$): isomer **3a**: δ = 1.82 (s, 6H, 11,11-di-CH_3); 8.50 (s, 1H, 3-H); 7.40–7.65 ppm (m, 5H, 6-C_6H_5); isomer **3b**: δ = 1.80 (s, 6H, 11,11-di-CH_3); 8.55 (s, 1H, 6-H); 7.40–7.65 ppm (m, 5H, 3-C_6H_5).

Separation of both isomers on silica gel was not successful, even by flash chromatography.

9,9-Dimethyl-6-hydrazino-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]-pyridazine (5b):

To a solution of 6-chloro-9,9-dimethyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**4a**; 222 mg, 1 mmol) in boiling ethanol (10 ml), a solution of hydrazine hydrate (80%; 0.2 ml) is added dropwise. The mixture is heated under reflux for 3 h. After cooling, the precipitate is isolated by suction and recrystallized from ethanol; yield: 164 mg (75%); m.p. 224–227 °C.

$C_8H_{10}N_8$	calc.	C 44.03	H 4.62	N 51.35
(218.2)	found	44.27	4.98	51.07

^{1}H-N.M.R. (DMSO-d_6/TMS$_{int}$): δ = 1.67 (s, 6H, 9,9-di-CH_3); 4.42 (br. s, 2H, NH_2); 9.30 (br. s, 1H); 9.25 ppm (s, 1H, 3-H).

6-Benzylidenehydrazino-9,9-dimethyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo-[4,3-*b*]pyridazine (5c):

To a hot, stirred solution of 9,9-dimethyl-6-hydrazino-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**5b**; 218 mg, 1 mmol) in methanol (20 ml), a solution of benzaldehyde (136 mg) in methanol (5 ml) is added dropwise and the mixture is heated under reflux for 5 h. After cooling, the precipitate is isolated by suction and recrystallized from ethanol; yield: 275 mg (90%); m.p. 286–288 °C.

$C_{15}H_{14}N_8$	calc.	C 58.81	H 4.61	N 36.58
(306.3)	found	58.92	4.61	36.77

^{1}H-N.M.R. (DMSO-d_6/TMS_{int}): δ = 1.70 (s, 6H, 9,9-di-CH_3); 3.78 (s, 1H, CH=N); 7.1–7.7 (m, 5H_{arom}); 8.28 (s, 1H, NH); 9.03 ppm (s, 1H, 3-H).

11,11-Dimethyl-6-phenyl-11*H*-pyrazolo[3,4-*d*]bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine (3a):

To a stirred mixture of 6-benzylidenehydrazino-9,9-dimethyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**5c**; 306 mg, 1 mmol) and sodium acetate (0.5 g) in glacial acetic acid (5 ml), a solution of bromine (≈ 200 mg) in glacial acetic acid (5 ml) is added dropwise at room temperature. The mixture is stirred for another 3 h at room temperature, then diluted with water (20 ml) and extracted with chloroform (3 × 10 ml). The combined extracts are dried with sodium sulphate, the solvent is evaported in vacuo; and the dry residue is recrystallized from ethanol; yield: 260 mg (85%); m.p. 255–257 °C.

$C_{15}H_{12}N_8$	calc.	C 59.20	H 3.97	N 36.82
(304.3)	found	58.83	3.71	36.75

^{1}H-N.M.R. ($CDCl_3$/TMS_{int}): δ = 1.82 (s, 6H, 11,11-di-CH_3); 7.40–7.65 (m, 5H_{arom}); 8.50 ppm (s, 1H, 3-H).

6-Chloro-9,9-dimethyl-3-phenyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (5d):

This compound is prepared from 6-chloro-3-phenyl-*s*-triazolo[4,3-*b*]pyridazine (**4b**) in essentially the same way as described earlier for 6-chloro-9,9-dimethyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**5a**)[15]; yield: 85%; m.p. 265–270 °C (from dimethylformamide/chloroform).

$C_{14}H_{11}ClN_6$	calc.	C 56.29	H 3.71	N 28.13
(298.7)	found	56.45	3.97	28.40

^{1}H-N.M.R. (DMSO-d_6/TMS_{int}): δ = 1.75 (s, 6H, 9,9-di-CH_3); 7.35–7.60, 8.25 ppm (2 m, 5H_{arom}).

6-Hydrazino-9,9-dimethyl-3-phenyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4.3-*b*]pyridazine (5e):

To a solution of 6-chloro-9,9-dimethyl-3-phenyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**5d**; 298 mg, 1 mmol) in boiling ethanol (50 ml), hydrazine hydrate (80%; 0.2 ml) is added, and the mixture is heated under reflux for 3 h. The precipitate is isolated by suction and recrystallized from ethanol; yield: 205 mg (70%); m.p. 240–242 °C.

$C_{14}H_{14}N_8$	calc.	C 57.13	H 4.79	N 38.07
(294.3)	found	56.98	4.98	38.31

^{1}H-N.M.R. (DMSO-d_6/TMS_{int}): δ = 1.71 (s, 6H, 9,9-di-CH_3); 4.55 (br. s, 2H, NH_2); 7.35–7.60, 8.35–8.60 (2 m, 5H_{arom}); 9.45 ppm (br. s, 1H, NH).

11,11-Dimethyl-3-phenyl-11*H*-pyrazolo[3,4-*d*]bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine (3b):

A stirred mixture of 6-hydrazino-9,9-dimethyl-3-phenyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**5e**; 294 mg, 1 mmol) and diethoxymethyl acetate (1.5 ml) is heated at 80 °C for 5 h. After cooling, the solid product is isolated by suction and recrystallized from methanol; yield: 152 mg (50%); m.p. 264–266 °C.

$C_{15}H_{12}N_8$	calc.	C 59.20	H 3.97	N 36.82
(304.3)	found	59.08	3.91	36.64

^{1}H-N.M.R. (DMSO-d_6/TMS_{int}): δ = 1.80 (s, 6H, 11,11-di-CH_3); 7.73 (br. s, 10H_{arom}); 8.55 ppm (s, 1H, 6-H).

6-Benzylidenehydrazino-9,9-dimethyl-3-phenyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (5f):

To a solution of 6-hydrazino-9,9-dimethyl-3-phenyl-9*H*-pyrazolo-[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**5e**; 294 mg, 1 mmol) in boiling ethanol (50 ml), a solution of benzaldehyde (136 mg) in ethanol (5 ml) is added dropwise. The mixture is then heated under reflux for 1 h. The solvent is evaporated in vacuo and the residue recrystallized from ethyl acetate; yield: 363 mg (95%); m.p. 225–227 °C.

$C_{21}H_{18}N_8$	calc.	C 65.96	H 4.74	N 29.30
(382.4)	found	65.71	5.02	29.07

^{1}H-N.M.R. (DMSO-d_6/TMS_{int}): δ = 1.75 (s, 6H, 9,9-di-CH_3); 7.25–7.80, 8.40–8.70 (2 m, 10H_{arom}); 8.48 (s, 1H, CH=N); 11.2 ppm (br. s, NH).

11,11-Dimethyl-11*H*-pyrazolo[3,4-*d*]bis-*s*-triazolo[4,3-*b*:3′,4′-*f*]pyridazine (6):

A mixture of 6-hydrazino-9,9-dimethyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**5b**; 218 mg, 1 mmol) and diethoxymethyl acetate (1,5 ml) is heated under reflux for 30 min. After cooling, the precipitate is isolated by suction and recrystallized from water; yield: 160 mg (70%); m.p. > 300 °C.

$C_9H_8N_8$	calc.	C 47.37	H 3.53	N 49.10
(228.2)	found	47.37	3.55	48.91

^{1}H-N.M.R. (DMSO-d_6/TMS_{int}): δ = 1.75 (s, 6H, 11,11-di-CH_3); 1.13, 1.15 ppm (2 s, 1H each, 3-H, 6-H).

11,11-Dimethyl-3,6-diphenyl-11*H*-pyrazolo[3,4-*d*]bis-*s*-triazolo-[4,3-*b*:3′,4′-*f*]pyridazine (7):

To a stirred solution of 6-benzylidenehydrazino-9,9-dimethyl-3-phenyl-9*H*-pyrazolo[4,3-*d*]-*s*-triazolo[4,3-*b*]pyridazine (**5f**; 382 mg, 1 mmol) and sodium acetate (300 mg) in glacial acetic acid (10 ml), a solution of bromine (10% in glacial acetic acid) is added dropwise until all bromine has been consumed. The solution is then poured onto crushed ice (40 g). The precipitated product is isolated by suction and recrystallized from methanol; yield: 190 mg (50%); m.p. 306–311 °C.

$C_{21}H_{16}N_8$	calc.	C 66.30	H 4.24	N 29.46
(380.4)	found	66.23	4.27	29.46

^{1}H-N.M.R. ($CDCl_3$/TMS_{int}): δ = 1.90 (s, 6H, 11,11-di-CH_3); 6.90–7.20 ppm (m, 10H_{arom}).

The authors thank the Research Community of Slovenia for financial support of this work and the Chemical Institute Boris Kidrič, Ljubljana, for a partial fellowship (B.F.)

We are grateful to Dr. K.L. Loening, Nomenclature Director, Chemical Abstracts Service, for helping to correctly name, number, and arrange the new heterocyclic system.

Received: February 21, 1985

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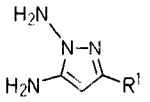
Errata and Addenda 1986

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, 2 H_{arom}); 7.46 (d, 2 H_{arom}); 7.3 (s, 5 H_{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, 5 H_{arom}); 7.3 (s, 5 H_{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).

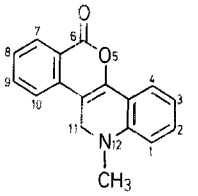
The ^{1}H-NMR data for compound **6** (p. 54) should be:
^{1}H-NMR ($CDCl_3$/TMS_{int}): δ = 8.03 (d, 2 H_{arom}); 7.43 (d, 2 H_{arom}); 5.65 (s, 1 H, CH); 5.23 (s, 2 H, CH_2); 4.5 (s, 1 H, NH); 1.53, 1.35 ppm (2 s, 6 H, 2 CH_3).

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:

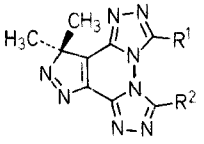


O. Meth-Cohn *Synthesis* **1986**, 76. The correct numbering for compounds **8** and **10** (p. 76) is as illustrated below for compound **10**:



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:

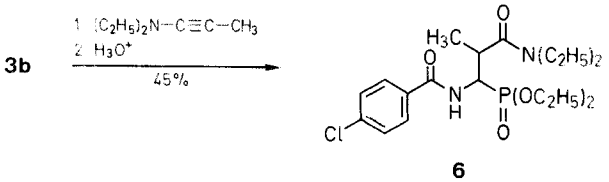


N. Petragnani, H. M. C. Ferraz, G. V. J. Silva *Synthesis* **1986**, 157. The authors wish to include the following pertinent references:
R. M. Adlington, A. G. M. Barret *Tetrahedron* **1981**, *37*, 3935.
R. M. Adlington, A. G. M. Barret *J. Chem. Soc. Perkin Trans. I* **1981**, 2848.
R. M. Adlington,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

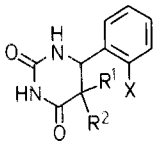
D. P. Matthews, J. P. Whitten, J. R. McCarthy *Synthesis* **1986**, 336. The headings for the first and last experimental procedures should be, respectively:
***N*^{1},*N*^{3}-Bis(2,2-dimethoxyethyl)oxaldiamidine Dihydrochloride (2):**
2-(2-Imidazolyl)-4-methoxy-4,5-dihydroimidazole (5):

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

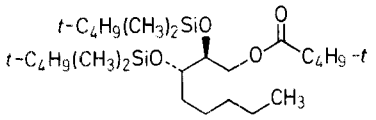


6

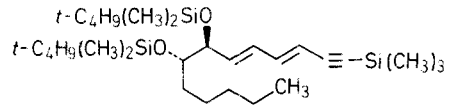
D. N. Dhar, K. S. K. Murthy *Synthesis* **1986**, 437. The heading for Table 2 (p. 440) should be:
4-Aryl-2(1*H*)-quinazolines **(13)** and 4-Aryl-1*H*-2,1,3-benzothiadiazine 2,2-Dioxides **(14)**
The names of compounds **13a** and **14a** in the experimental procedure on the same page should be corrected 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

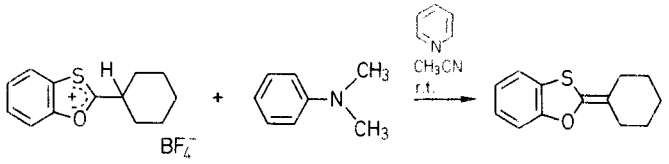


16

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:



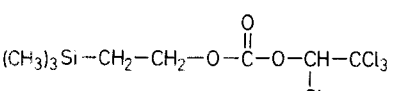
1c **2** **5**

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**