m/e 160 (p), 159 (p - 1), 144, 130, 118, 117, 116, 114, 104.

2-Ethyl-p-xylene: IR (neat) 2970, 2900, 1500, 1460, 1380, 1060, 800 cm^{-1}; ^1H NMR ($CDCl_3$) δ 6.98 (3 H, m), 2.59 (2 H, q, J = 7.5 Hz), 2.29 (3 H, s), 2.25 (3 H, s), 1.19 (3 H, t, 7.5 Hz); mass spectrum (70 eV), m/e 134 (p), 119 (p - CH_3), 105 (p - CH_2CH_3), 95, 91.

2-Methoxy-6-methyl-9,10-dihydrophenanthrene: IR (neat) 3000, 2850, 1600, 1490, 1460, 1425, 1300, 1270, 1230, 1150, 1100, 1030, 900, 840, 790, 720 cm^{-1}; ^1H NMR ($CDCl_3$) δ 6.99 (6 H, br m), 3.75 (3 H, s), 2.74 (4 H, s), 2.30 (3 H, s); mass spectrum (70 eV), m/e 224 (p) 223 (p - 1), 208 (p - 1 - CH_3), 193, 192, 178, 177, 165, 164.

(+)-Calamenene: IR (neat) 2960, 2880, 1440, 1370, 1370, 800 cm^{-1}; ^1H NMR ($CDCl_3$) 6.93 (3 H, br s), 2.23 (3 H, s), 1.23–0.60 (9 H); mass spectrum (70 eV), m/e 202 (p), 201 (p - 1), 158, 145, 144, 143, 142, 141, 130, 128, 127, 105.

Benzocyclododecene: IR (neat) 2920, 2850, 1480 cm^{-1}; ^1H NMR ($CDCl_3$) 7.13 (4 H, br s), 2.64 (4 H, t, J = 7.6 Hz), 1.52 (16 H, m).

Compound 6: ^1H NMR ($CDCl_3$) δ 9.95 (1 H, s), 5.43 (2 H, br s), 3.33 (2 H, br s), 0.87 (3 H, s), 0.07 (9 H, s); mass spectrum (70 eV), m/e 278 (p), 277 (p - 1), 263 (p - CH_3), 249, 221 (p - $(CH_3)_3C$), 205 (p - $(CH_3)_3Si$), 193, 191, 179, 130, 91, 75, 73 ($(CH_3)_3Si$).

Compound 7: ^1H NMR ($CDCl_3$) δ 7.07 (3 H, s), 2.78 (4 H, br m), 0.94 (9 H, s), 0.24 (9 H, s); mass spectrum (70 eV), m/e 260 (p), 245 (p - CH_3), 203 (p - $(CH_3)_3C$), 189 (p - $(CH_3)_3Si$), 132, 73 ($(CH_3)_3Si$).

Compound 9: IR (CH_2Cl_2) 2960, 1660, 1370, 830 cm^{-1}; ^1H NMR ($CDCl_3$) 2.17 (8 H, br m), 0.87 (9 H, s), 0.01 (9H, s); mass spectrum (70 eV), m/e 278 (p), 263 (p - CH_3), 221 (p - $(CH_3)_3C$), 195, 194, 193, 181, 180, 179, 147, 131, 91, 73 ($(CH_3)_3Si$).

Acknowledgment is made to the donors of he Petroleum Research Fund, administered by the American Chemical Society, for their generous support. We thank Lynette M. Gehring and Katherine S. Takaki for technical assistance and Lars Bergknut for obtaining mass spectra. S.A. is grateful to the East West Center for financial support.

Registry No. 3 (R = H), 81857-26-3; **3** (R = Me), 81857-27-4; **4** (R = Me), 81857-28-5; **5**, 81857-29-6; **6**, 81857-30-9; **7**, 81857-31-0; **8**, 42044-22-4; **9**, 81857-32-1; 7-isocalamenene, 483-77-2; methallyl chloride, 563-47-3; 2-(hydroxymethylene)cyclododecanone, 949-07-5; 2-[[(trimethylsilyl)oxy]methylene]cyclododecanone, 81857-33-2; 2-methylbenzocyclopentene, 874-35-1; 2-*tert*-butyl-6-methyl-1,2,3,4-tetrahydronaphthalene, 81857-34-3; 2-methylbenzocycloheptene, 827-40-7; 2-ethyl-*p*-xylene, 1758-88-9; 2-methoxy-6-methyl-9,10-dihydrophenanthrene, 81857-35-4; (+)-calamenene, 22339-23-7; benzocyclododecene, 7125-10-2; 2-(hydroxymethylene)cyclopentanone, 2-(hydroxymethylene)cyclopentanone; 4-*tert*-butyl-2-(hydroxymethylene)cyclohexanone, 22252-96-6; 2-(hydroxymethylene)cycloheptanone, 934-20-3; 1-hydroxy-2-methyl-1-penten-3-one, 50421-81-3; 6-methoxy-2-(hydroxymethylene)-1,2,3,4-tetrahydronaphthalenone, 16252-53-2; *cis*-2-(hydroxymethylene)-3-methyl-6-isopropylcyclohexanone, 59123-00-1; 2-[[(*tert*-butyldimethylsilyl)oxy]methylene]-cyclododecanone, 81857-36-5; *trans*-2-(hydroxymethylene)-3-methyl-6-isopropylcyclohexanone, 59122-99-5.

Biomimetic Synthesis of (±)-Pallescensin 1

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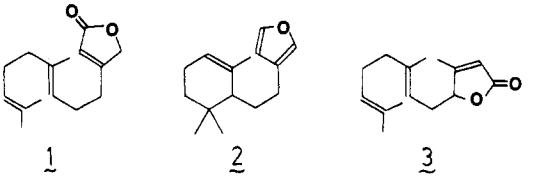
Received September 14, 1981

During an investigation of a biomimetic approach to the synthesis of the pallescensins[1] and the drimane sesquiterpenes,[2] we considered that butenolide 1 or its derivatives might serve as a common precursor to both classes of compounds. Cationic cyclization of the A ring would lead to (±)-pallescensin 1 (2) whereas bicyclization would furnish drimenin.[3] A recently published preparation of starting material 1 by oxidation of farnesal *O*-trimethylsilyl cyanohydrin appeared well suited to our purpose.[4] Farnesol,[5] available as a mixture of *E* and *Z* isomers at C-2, was oxidized to a mixture (ca. 1:1) of *E* and *Z* aldehydes. Conversion to the unstable *O*-trimethylsilyl cyanohydrins was accomplished by the use of trimethylsilyl cyanide and potassium cyanide/18-crown-6 complex.[6] Oxidation with pyridinium dichromate[4,7] in dry *N,N*-dimethylformamide gave a mixture of butenolides 1 and 3 in 62% overall yield from the aldehyde mixture. Proton NMR spectroscopy at 100 MHz gave no indication of the presence of butenolide isomers. Multiple TLC elutions failed to effect separation, but suggested the presence of two compounds. This was confirmed by reduction to a mixture of furans. Chromatographic separation of 1 and 3 was not practical on a preparative scale, so the separation was effected at the aldehyde stage.[8] Whereas the *E* aldehyde was oxidized to butenolide 1 regiospecifically, as shown by reduction with diisobutylaluminum hydride[9] to pure dendrolasin 4[10] (54% yield), treatment of the *Z* aldehyde under identical conditions furnished a reproducible 60:40 mixture of 4 and sesquirosefuran 5[11] (57% yield). The ratio of 4 to 5 in the product mixture was easily determined by integration of the signals for the furan β protons in the NMR spectrum. It is thus shown that the butenolide synthesis is regiospecific only for the *E* aldehyde.



1 2 3

The reaction of 1 with Lewis acids was subsequently examined. Treatment of 1 with 2 equiv of stannic chloride in dry dichloromethane at −30 °C for 1 h gave in 60% isolated yield the desired monocyclic butenolide 6,[12] ac-

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(2) Heathcock, C. H. In "The Total Synthesis of Natural Products"; J. W. ApSimon, Ed.; Wiley-Interscience: New York, 1973; Vol. 2, pp 338–353.

(3) Cation olefin cyclization reactions have been reviewed extensively: Johnson, W. S. *Acc. Chem. Res.* **1968**, *1*, 1. Johnson, W. S. *Angew. Chem., Int. Ed. Engl.* **1976**, 15, 9. Johnson, W. S. *Bioorg. Chem.* **1976**, *5*, 51.

(4) Corey, E. J.; Schmidt, G. *Tetrahedron Lett.* **1980**, 731. Other syntheses of 1 have been reported: (a) Kobayashi, M.; Negishi, E. *J. Org. Chem.* **1980**, *45*, 5223. (b) Takahashi, S. *Synth. Commun.* **1976**, 6, 331.

(5) A gift of farensol from the Givaudan Corp., Clifton, NJ, is gratefully acknowledged.

(6) (a) Evans, D. A.; Truesdale, L. K. *Tetrahedron Lett.* **1973**, 4929. (b) Parham, W. E.; Roosevelt, C. S. *Ibid.* **1971**, 923. (c) Evans, D. A.; Truesdale, L. K.; Carroll, G. L. *J. Chem. Soc., Chem. Commun.* **1973**, 55. (d) Evans, D. A.; Carroll, G. L.; Truesdale, L. K. *J. Org. Chem.* **1974**, 39, 914.

(7) Corey, E. J.; Schmidt, G. *Tetrahedron Lett.* **1979**, 399.

(8) The undesired *Z* aldehyde was converted to an isomeric mixture of aldehydes by treatment at 23 °C with powdered potassium carbonate in methanol. This allowed recycling of the unwanted isomer.

(9) (a) Minato, H.; Nagasaki, T. *J. Chem. Soc. C* **1966**, 377. (b) Grieco, P. A.; Pogonowski, C. S.; Burke, S. *J. Org. Chem.* **1975**, 40, 542. (c) McMurry, J. E.; Donovan, S. F. *Tetrahedron Lett.* **1977**, 2869.

(10) For earlier syntheses of dendrolasin, see: (a) Parker, K. A.; Johnson, W. S. *Tetrahedron Lett.* **1969**, 1329. (b) Thomas, A. F. *J. Chem. Soc., Chem Commun.* **1968**, 1957. (c) Garst, M. E.; Spencer, T. A. *J. Am. Chem. Soc.* **1973**, *95*, 250. (d) Still, W. C. *Ibid.* **1978**, *100*, 1481. (e) Kondo, K.; Matsumoto, M. *Tetrahedron Lett.* **1976**, 391.

(11) Isolation and structure elucidation: Hayashi, N.; Komae, H.; Eguchi, S.; Nakayama, M.; Hayashi, S.; Sakao, T. *Chem. Ind. (London)* **1972**, 572. Synthesis: Gedge, D. R.; Pattenden, G. *Tetrahedron Lett.* **1977**, 4443. Takahashi, Y.; Hagiwara, H.; Uda, H.; Kosugi, H. *Heterocycles* **1981**, *15*, 225.

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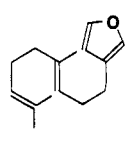

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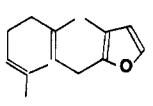

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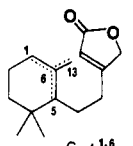
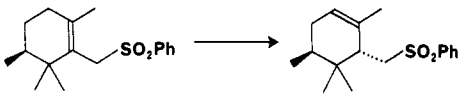


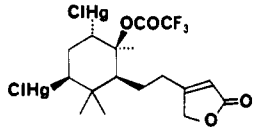
6 $\Delta^{1,6}$
7 $\Delta^{6,13}$
8 $\Delta^{5,6}$

companied by ca. 10% of exocyclic isomer **7**,[12] and a trace of tetrasubstituted olefin **8**.[12] Longer reaction times and higher temperatures led to increasing amounts of **8** in the product mixture. When cyclization was allowed to take place at 0 °C, isomer **8** was the only observed product. Further warming to 23 °C led to no change in the reaction product. It thus appears that preferential kinetic deprotonation at C-1 (pallescensin numbering) of the putative tertiary carbonium ion intermediate takes place, followed by slow (at −30 °C) acid-catalyzed isomerization to the tetrasubstituted product. It is noteworthy that in a closely related system the opposite trend was observed: isomerization of sulfone **9** to the trisubstituted isomer **10** was observed in the presence of acid.[13]



9 10

The preference for **8** at higher temperatures was observed with other Lewis acids. For example, treatment of **1** with boron trifluoride etherate in benzene for 2.5 h at reflux gave **8** as the major product. Further evidence of the kinetic preference for **6** was also forthcoming. The use of an excess of mercuric trifluoroacetate[14] in nitromethane at −25 °C initially furnished the $\Delta^{1,6}$ isomer as the major product. Subsequent trapping by a second equivalent of mercuric salt, followed by workup with aqueous sodium chloride gave crystalline bis(chloromercury) trifluoroacetoxy compound **11** (61% yield).



11

The conversion of butenolide **1** to (±)-pallescensin **1**, identical by spectroscopic comparison with authentic material,[15] was accomplished by treatment in tetrahydrofuran with 1.3 equiv of diisobutylaluminum hydride.[9] The conversion of **1** to drimenin or isodrimenin is being investigated.

(12) Butenolide **6**: IR (neat) 2950, 1785, 1760, 1640, 1450, 1170, 1130, 1020, 880, 850 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.85 (t, J = 1.6 Hz, 1 H), 5.40 (br s, 1 H), 4.75 (d, J = 1.6 Hz, 2 H), 1.69 (overlapping br d, 3 H), 0.95 (s, 3 H), 0.90 (s, 3 H). Butenolides **7** and **8** were readily distinguished by ^1H NMR at 100 MHz. For **7** signals at δ 4.50 and 4.80, due to the exocyclic methylene group, were diagnostic. **8** was identified by the absence of a second olefinic signal and the collapse of the aliphatic methyl peaks to a singlet at δ 0.98.

(13) Torii, S.; Uneyama, K.; Matsunami, S. *J. Org. Chem.* **1980**, 45, 16.

(14) The efficacy of mercuric trifluoroacetate as an initiator for cationic cyclizations has been demonstrated: (a) Kurbanov, M.; Semenovsky, A. V.; Smit, W. A.; Shmelev, L. V.; Kucherov, V. F. *Tetrahedron Lett.* **1972**, 2175. (b) Skeean, R. W.; Trammell, G. L.; White, J. D. *Ibid.* **1976**, 525. (c) Corey, E. J.; Tius, M. A.; Das, J. *J. Am. Chem. Soc.* **1980**, *102*, 1742. (d) Corey, E. J., Tius, M. A.; Das, J. *Ibid.* **1980**, *102*, 7612.

(15) We are grateful to Dr. G. Cimino and Dr. T. Matsumoto for infrared and proton NMR spectra of pallescensin 1.

Experimental Section

Tetrahydrofuran was distilled from sodium benzophenone ketyl, methylene chloride was distilled from phosphorus pentoxide, and nitromethane was distilled from calcium hydride immediately prior to use. Stannic chloride was distilled from mossy tin. Nuclear magnetic resonance (NMR) spectra were recorded at 100 MHz on a Varian XL-100 spectrometer. Chemical shifts are reported in parts per million (δ) relative to tetramethylsilane. Infrared (IR) spectra were recorded either on a Beckman IR 10 or a Perkin-Elmer 410 A. Mass spectra were recorded on a Varian MAT-311 instrument.

Chloromercurial 11. A solution of 10 mg of butenolide **1** in 180 μL of anhydrous nitromethane was chilled to −25 °C. A solution of 25 mg (1.4 equiv) of mercuric trifluoroacetate dissolved in the minimum amount of the same solvent was added to the stirred solution of the butenolide via cannula. After 20 min the reaction mixture was poured onto a saturated aqueous solution of sodium chloride and was stirred for ca. 10 min. The aqueous phase was extracted thoroughly with methylene chloride. The organic extracts were washed with saturated aqueous sodium bicarbonate, followed by water and brine. Drying ($MgSO_4$) and solvent evaporation furnished the crude product which was purified by column chromatography on silica gel to produce 21 mg (61% yield) of solid organomercurial **11**:[16] IR IR 2960, 1785, 1760, 1640, 1130 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.86 (t, J = 1.4 Hz, 1 H, C=CH), 4.75 (d, J = 1.4 Hz, 2 H, CH_2O), 2.72 (m, 4 H, ClHgCH and C=CCH_2), 1.23 (s, 3 H, CH_3), 1.14 (s, 3 H, CH_3), 1.07 (s, 3 H, CH_3).

Butenolide 6. A solution of 20 mg of butenolide **1** in 430 μL of anhydrous dichloromethane was maintained between −30 and −40 °C while 20 μL (2 equiv) of stannic chloride was added. After 1 h the reaction mixture was poured onto saturated aqueous sodium bicarbonate and ice. Filtration, followed by extraction with methylene chloride, drying (K_2CO_3), and solvent evaporation furnished the crude product which contained, in addition to **6**, small amounts of **7** and **8**. A pure sample of **6** (12 mg, 60% yield) was obtained by chromatography on silica gel: IR (neat) 2950, 1785, 1760, 1640, 1450, 1170, 1130, 1020, 880, 850 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.85 (t, J = 1.6 Hz, 1 H, butenolide C=CH), 5.40 (br s, 1 H, C=CH), 4.75 (d, J = 1.6 Hz, 2 H, CH_2O), 1.69 (overlapping br d, 3 H, C=CCH_3), 0.95 (s, 3 H, CH_3), 0.90 (s, 3 H, CH_3); mass spectrum (70 eV), m/e 234 (p), 219 (p − CH_3), 179, 178, 165, 149, 148, 136, 123, 108, 106, 98.

(±)-Pallescensin 1 (2). A solution of 28 mg of butenolide **6** in 1.2 mL of anhydrous tetrahydrofuran was treated with 156 μL (1.3 equiv) of a 1 M solution of diisobutylaluminum hydride in hexane at −20 °C. After 1 h at this temperature acetic acid was added and the reaction mixture was allowed to warm to 23 °C. The heterogeneous solution was diluted with methylene chloride and centrifuged to separate the solid materials. The supernatant was removed and the solid resuspended with methylene chloride and centrifuged. The combined organic phase was washed with saturated aqueous sodium chloride, dried (K_2CO_3), and purified by column chromatography on silica gel to give 13 mg (50% yield) of (±)-pallescensin 1, identical by IR and NMR comparison with authentic material:[15] IR (neat) 2950, 2850, 1500, 1450, 1380, 1360, 1160, 1130, 1060, 1010, 880 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.24 (br s, 1 H, Ar H), 7.19 (br s, 1 H, Ar H), 6.20 (br s, 1 H, Ar H), 5.28 (br s, 1 H, C=CH), 1.66 (br s, 3 H, C=CCH_3), 0.92 (s, 3 H, CH_3), 0.86 (s, 3 H, CH_3); mass spectrum (70 eV), m/e 218 (p), 203 (p − CH_3), 162, 147, 133, 123, 109, 95, 81.

Acknowledgment. The generous support of the Research Corporation is gratefully acknowledged. A Biomedical Research Support Grant, administered by the University of Hawaii, is also acknowledged. We thank Mr. Sadaquat Ali for experimental assistance.

Registry No. (*E*)-**1**, 61315-76-2; (±)-**2**, 82043-26-3; (*E*)-(±)-**3**, 82010-08-0; (*E*)-**4**, 23262-34-2; (*E*)-**5**, 39007-93-7; (±)-**6**, 82025-97-6; (±)-**7**, 82010-09-1; **8**, 78816-32-7; **11**, 82010-10-4; (*E*,*Xc*)-farnesol,

(16) We were unable to record a satisfactory mass spectrum or melting point for this compound.

(17) Undergraduate researcher.

82010-11-5; (*Z*,*X*)-farnesol, 82010-12-6; (*E*,*X*)-farnesal, 80442-43-9; (*Z*,*X*)-farnesal, 80442-44-0; (*E*,*X*)-farnesal *O*-trimethylsilyl cyanohydrin, 82025-98-7; (*Z*,*X*)-farnesal *O*-trimethylsilyl cyanohydrin, 82025-99-8.

200-MHz Proton Nuclear Magnetic Resonance Study of the Naphtho[2,1-*e*]tetrazolo[5,1-*c*]-*as*-triazine/3-Azidonaphtho[2,1-*e*]-*as*-triazine/Naphtho[2,1-*e*]tetrazolo[1,5-*b*]-*as*-triazine Equilibrium

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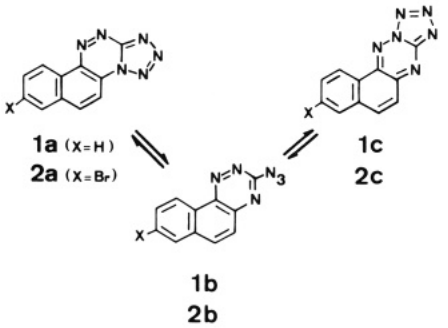
Received December 1, 1981

In the first paper of this series[1] the cyclization of several (2-hydroxynaphthalenyl)-1-azoazoles to naphtho[2,1-*e*]-azolo-*as*-triazines was reviewed and investigated, the structure of most of these tetracyclic compounds being firmly established on the basis of the common spectroscopic techniques. However, with regard to the corresponding tetrazole derivative, naphtho[2,1-*e*]tetrazolo-[5,1-*c*]-*as*-triazine (**1a**), which may tautomerize to 3-azi-



donaphtho[2,1-*e*]-*as*-triazine (**1b**) and naphtho[2,1-*e*]tetrazolo[1,5-*b*]-*as*-triazine (**1c**),[2,3] doubt still remained: whereas, as shown by IR spectroscopy, the azide form (**1b**) seemed to predominate in $CHCl_3$, we had inconclusive proof concerning the true structure (**1a**, **1c**, or a mixture of both) of the cyclic, major product(s) in the solid state and in Me_2SO solution at room temperature. An easy approach to the question would have been to perform an NMR study of **1** in suitable solvents at about 20 °C, but the low solubility of **1** precluded it at that moment.

We report here a 200-MHz FT ^1H NMR study of the 8-bromo derivative of **1** (**2**), a more appropriate compound than **1** for analytical purposes, in Me_2SO-d_6 and in $CDCl_3$, which establishes without doubt that in the former solvent the only tautomer present is **2c** while in the latter there is an equilibrium between **2b** and **2c**.

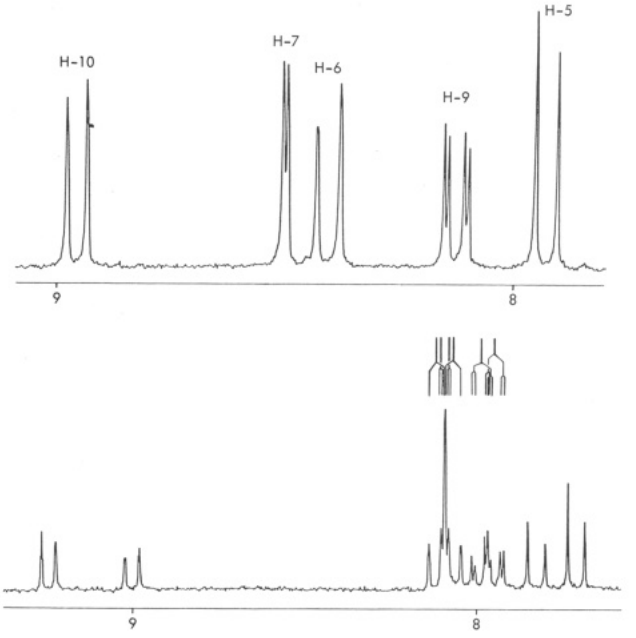
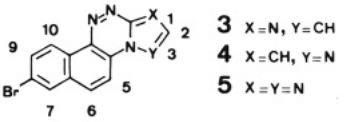


Figure 1. 200-MHz ^1H NMR spectrum of **2** in Me_2SO-d_6 (top) and in $CDCl_3$ (bottom).

The preparation of **2** was accomplished as in the case of **1**,[1] i.e., by coupling diazotetrazole with the corresponding 2-naphthol and refluxing the azo derivative under acidic conditions. For the sake of comparison, 8-bromonaphtho[2,1-*e*]imidazo[2,1-*c*]-*as*-triazine (**3**), 8-bromonaphtho[2,1-*e*]pyrazolo[5,1-*c*]-*as*-triazine (**4**), and 8-bromonaphtho[2,1-*e*]-*s*-triazolo[5,1-*c*]-*as*-triazine (**5**) were similarly obtained.



The ^1H NMR spectrum of **2** in Me_2SO-d_6 at 20 °C, practically a first-order spectrum, is shown in Figure 1 (top). It is obvious that a single tautomer is present. Furthermore, if the observed chemical shifts are compared with those of the protons of **3**–**5** in the same solvent (see Table I), it will be noted that H-5 of **2** appears at higher field than the analogous protons of **3**–**5** (the complex signal including H-5, H-6, and H-7 lies at ca. δ 8.5 in all cases), and the δ value for H-10 of **2** is lower than expected. Therefore, it can be deduced that the tautomer of **2** that largely predominates under those conditions is not **2a**.[4] Taking into account that the azide band is absent from the IR spectrum of **2** in Me_2SO (and in the solid state[5]), we can conclude that the "true structure" of **2** must be **2c**.

The ^1H NMR spectrum of **2** in $CDCl_3$ at 20 °C, shown in Figure 1 (bottom), affords an indirect confirmation of the above statement. Two sets of signals are apparent, which can be attributed to **2b** (57%) and **2c** (43%)[6] in view of the strong azide bands present in the IR spectrum in $CHCl_3$ and the δ values observed for the H-5 and H-10

(1) Vilarrasa, J.; Granados, R. *J. Heterocycl. Chem.* **1974**, *11*, 867.
(2) For reviews on the azide–tetrazole equilibrium, see, e.g.: (a) Butler, R. N. *Adv. Heterocycl. Chem.* **1977**, *21*, 323. (b) Tisler, M. *Synthesis* **1973**, 123.
(3) The existence of a similar ternary equilibrium in the benzo-*as*-triazine series has been recently demonstrated: (a) Messmer, A.; Hajós, G.; Tamás, J.; Neszmélyi, A. *J. Org. Chem.* **1979**, *44*, 1823. (b) Castillón, S.; Meléndez, E.; Pascual, C.; Vilarrasa, J., *J. Org. Chem.*, in press.
(4) The low δ value for H-5 agrees with structures **2b** and **2c**, in which no azole ring is close to that proton, as observed in naphtho[2,1-*e*]-*v*-triazolo[1,5-*b*]-*as*-triazine.[1] Protons H-5, H-6, and H-10 of a related compound, naphtho[2,1-*e*]-*as*-triazin-3-one, appear at δ 7.20, 8.16, and 8.52, respectively, in Me_2SO-d_6 (Lalor, F. J.; Scott, F. L.; Ferguson, G.; Marsh, W. C. *J. Chem. Soc., Perkin Trans. 1* **1978**, 789).
(5) Attempts to obtain crystals of **2** good enough for a X-ray analysis have been unsuccessful.
(6) On addition of TFA to this solution the **2b**/**2c** ratio increases, as expected.[2]

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