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Palladium-Catalyzed Allylation of 5-Membered Heterocyclic Ambident Sulfur Nucleophiles

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Abstract.- Pd(0)-Catalyzed allylation of five-membered ambident heterocycles bearing NH-CO and NH-CS moieties obey the regioselectivity rules N>O, S>N, NH-CO>NH-CS

INTRODUCTION

The Pd(0)-catalyzed allylation of heterocyclic systems bearing ambident nucleophiles is a topic of increasing interest Thus, regioselective N-9 allylation of purines, at the imidazole part of the molecule, is a key step in the preparation of carbanucleosides [1-7] Other ambident heterocyclic 5-membered rings possessing a tautomeric or mesomeric aromatic structure that have been allylated under Pd(0) catalysis include imidazole,[2,8] indole (C-3 allylation under thermodynamic control and N-allylation under kinetic control),[9,10] tetronic acids (C-allylation under thermodynamic control),[11] and ascorbic acid (vitamin C) (C-allylation) [12]

On the other hand, sulfur nucleophiles are not popular in Pd(0)-catalyzed allylation chemistry, possibly due to the belief that the pronounced thiophilicity of palladium could poison the catalytic systems However, Trost and Scanlan have described a Pd(0)-catalyzed synthesis of allyl sulfides[13] and also two scattered examples of allylations at sulfur under Pd[14] and Ni[15] catalysis have been reported

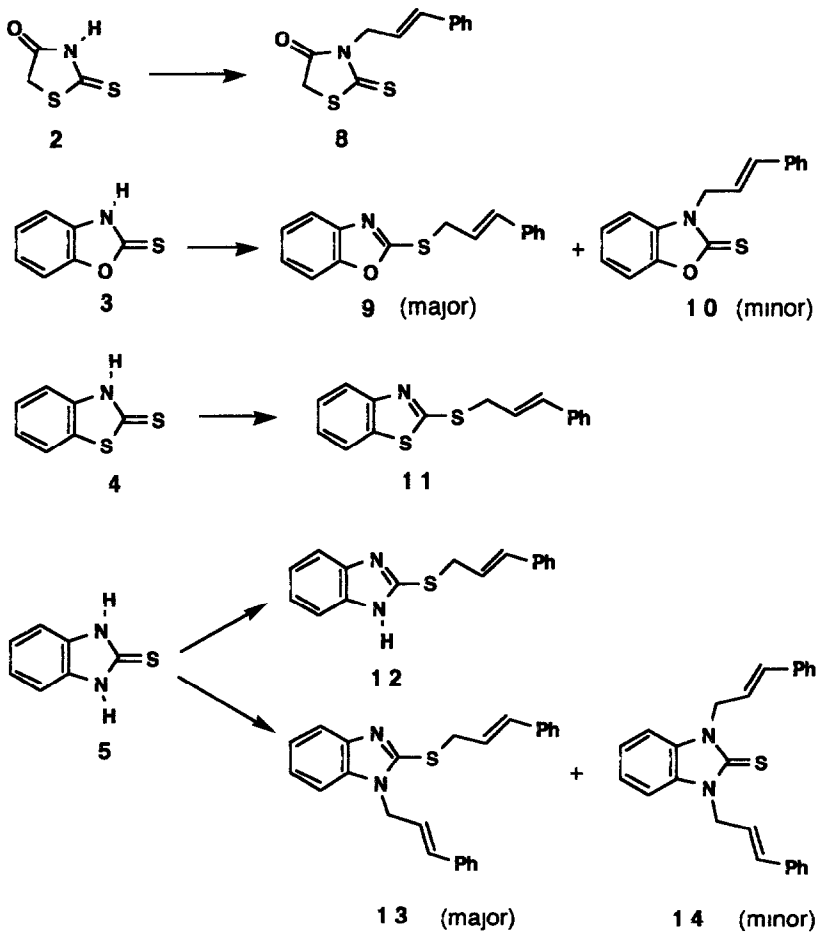
RESULTS

We have studied the Pd-catalyzed allylation of several 5-membered heterocyclic ambident systems bearing a nucleophilic sulfur atom, i e 2-thioxothiazolidin-4-one (rhodanine), **2**, thiobenzoxazolone, **3**, thiobenzothiazolone, **4**, thiobenzimidazolone, **5**, and 3(5)-mercapto-1,2,4-triazole, **7** Also, the related 1,2,4-triazole, **6**, is included in our study Cinnamyl ethyl carbonate, **1a**, has been selected for most of our experiments for its high regioselectivity

Our results are collected in the table and in the scheme Rhodanine, **2**, bears an amide and a thioamide group Its reaction with **1a** affords only product **8**, derived from allylation at the nitrogen atom (Run 1) Compounds **3-5** all possess the thioamide group, and in all cases (Runs 2-4) sulfur is allylated to afford products **9**, **11** and **12** When two equivalents of **1a** are introduced, as in run 5, the allylation occurs at sulfur and in one nitrogen atom (product **13**) rather than at both nitrogen atoms (product **14**) With no exception the regioselection rule is S>N

We wanted to study 3(5)-mercapto-1,2,4-triazole, **7**, and before that we selected 1,2,4-triazole, **6**, for preliminary studies Triazole **6** was efficiently allylated with several mixed allyl ethyl carbonates, **1a-d**, to afford compounds **15a-d** (Runs 6-10) In all cases reaction took place at N-1 as normally occurs under non metal catalyzed conventional alkylation conditions Mercaptotriazole **7**, when treated with 1 5 equivalents of **1a**, afforded **16** as the main product (Run 11) The treatment of **7** with two equivalents of **1a** produced a

mixture of compounds allylated at both S and N nucleophilic centers, namely **17** and **18** Again, a high regioselectivity favouring sulfur over nitrogen centers is observed

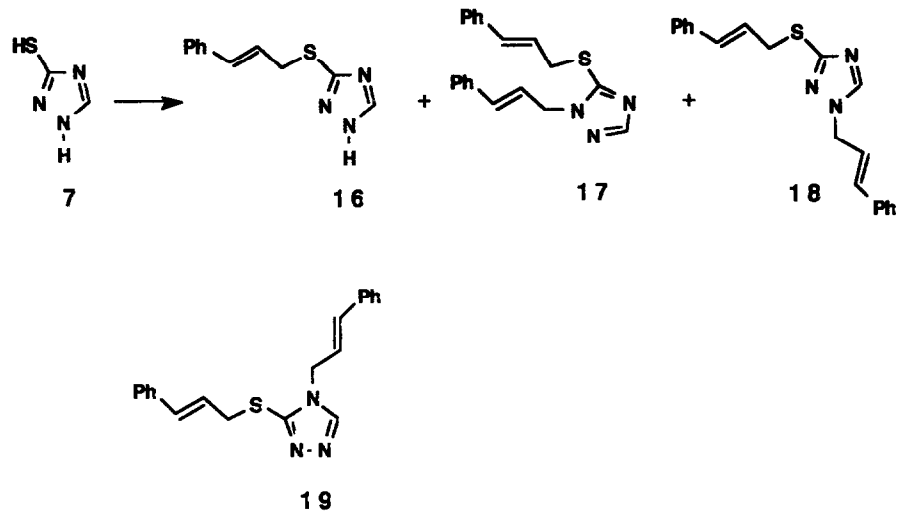


a R = Ph-CH=CH-CH_2-

b 

c CH_2=CH-CH_2-

d CH_2=C(CH_3)-CH_2-



Scheme For experimental conditions see table

Table - Allylation of Compounds **2-7** with allyl ethyl carbonates **1a-d**

Run	**2-7** (mmole)	**1** (mmole)	Pd[a] (mmole)	THF (mL)	temp	time	Product (%)	Allylation
1	**2**(10 0)	**1a**(12 0)	A(0 5)	25	rt	16h	**8**(63)	N > O NHCO > NHCS
2	**3**(7 0)	**1a**(7 0)	B(0 35)	25	rt	16h	**9**(76), **10**(3)	S > N
3	**4**(7 0)	**1a**(7 0)	B(0 35)	25	rt	16h	**11**(52)	S > N
4	**5**(7 0)	**1a**(7 0)	B(0 35)	25	rt	16h	**12**(36)	S > N
5	**5**(7 0)	**1a**(14 0)	B(0 35)	25	rt	24h	**13**(52), **14**(7)	S > N
6	**6**(14 2)	**1a**(28 4)	A(0 7)	50	Reflux	15h	**15a**(47)	N-1 > N-4
7	**6**(14 0)	**1b**(15 0)	A(0 84)	45	Reflux	96h	**15b**(22)	N-1 > N-4
8	**6**(7 2)	**1b**(7 7)	B(0 38)	40	Reflux	15h	**15b**(78)	N-1 > N-4
9	**6**(14 5)	**1c**(16 6)	B(0 72)	45	Reflux	3h	**15c**(81)	N-1 > N-4
10	**6**(7 2)	**1d**(8 9)	B(0 36)	30	Reflux	3h	**15d**(86)	N-1 > N-4
11	**7**(9 9)	**1a**(15 0)	B(0 46)	25	Reflux	3h	**16**(36), **17**(20), **18**(18)	S > N, N-1 = N-2
12	**7**(4 9)	**1a**(11 0)	B(0 25)	30	Reflux	3h	**17**(47), **18**(34)	N-1 = N-2

a A $Pd(acac)_2/PPh_3$ (1 4), B $Pd(PPh_3)_4$

Sulfide **12** was recovered after refluxing for 48 h in THF in the presence of $Pd(PPh_3)_4$ More interesting, the N-cinnamylthioamide **14** was also recovered after the same treatment These facts suggest that the major reactivity of S over N nucleophilic centers is kinetically controlled

All N-cinnamyl derivatives present a doublet at d 4 8-5 2, whereas S-cinnamyl derivatives exhibit the doublet at 3 9-4 2 Furthermore, the signals for the S-$\underline{CH_2}$ appear in 13C-NMR at 34-36 whereas those for N-$\underline{CH_2}$ appear above 45 Assignments of structures when regioisomers based on two different nitrogen atoms are possible, were made on the basis of the 13C-NMR spectra with the program Selective Distorsionless Enhancement by Polarization Transfer (SDEPT) developed by Sánchez-Ferrando and coworkers in our Department [16] As an example, by selective pulsing of the S- and the N- methylene protons of compound **17** only the coupled C-5 carbon atom (apart from the cinnamyl olefinic carbon atoms) showed its signal enhanced by polarization transfer (SDEPT effect) Also, by pulsing of the N-CH_2 protons of compound **18** only the coupled C-5 showed positive SDEPT effect Should the isomeric structure **19** have been the real one, SDEPT effect for signals due to both ring carbon atoms would have been observed A similar technique (Selective INEPT) has been recently reported [17]

FINAL REMARCK

When this work was already finished Prof Denis Sinou informed us that he and his coworkers had obtained results similar to those here described We are indebted to him for this communication [20]

EXPERIMENTAL

3-Cinnamyl-2-thioxothiazolidin-4-one, **8** (Run 1) (General procedure) A degassed solution of $Pd(acac)_2$ (0 152 g, 0 5 mmole), triphenylphosphine (0 525 g, 2 0 mmole) and cinnamyl ethyl carbonate, **1a**, (2 475 g, 12 0 mmole) in anhydrous THF (10 mL) was poured under argon into a degassed solution of 2-thioxothiazolidin-4-one (1 332 g, 10 0 mole) A yellow solid was formed The mixture was stirred for 16 h and the solid filtered off m p 229-230C (d), IR(KBr) 1714 cm^{-1} Anal Calcd for $C_{42}H_{34}N_2O_2P_2PdS_4$ = $((C_3H_2NOS_2)_2Pd(P(C_6H_5)_3)_2)$ C, 56.34; H, 3 83, N, 3 13, S, 14 32 Found C, 56 28, H, 3.78, N, 3 20, S, 14 32 The filtrate was evaporated and the residue was chromatographed through a column of silica-gel to afford **8** (1 571 g, 63%), m p 90-1C (diethyl ether), IR(KBr)· 1726 cm^{-1}; 1H-NMR ($CDCl_3$) 4 00 (s, 2H), 4 76 (d, J = 6 5 Hz, 2H), 6 18 (dt, J = 16 3 and 6 5 Hz, 1H), 6 74 (d, J = 16 3, 1H), 7 34 (m, 5H), 13C-NMR ($CDCl_3$) 35 2, 45 9, 120 4, 126 3, 127 9, 128.3, 135 2, 135 8, 173 2, 200 5, MS(m/e) 249(M, 11), 158(100), 115(68), 91(21) Anal Calcd for $C_{12}H_{11}NOS_2$ C, 57 80, H, 4 45, N, 5 62, S, 25 72 Found C, 57 58, H, 4 37, N, 5 63, S, 25 63

2-(Cinnamylthio)benzoxazole, **9**, and 3-cinnamylthiobenzoxazolone, **10** (Run 2) These compounds were obtained as for **8** After filtration of a Pd complex, the solution was evaporated and the residue chromatographed to afford **9** oil contaminated with a carbonyl containing impurity, 1H-NMR($CDCl_3$) 4 12 (d, J = 6 6 Hz, 2H), 6 35 (dt, J = 15 4 and 6 6 Hz, 1H), 6 75 (d, J = 15 4 Hz, 1H), 7 1-7 8 (m, 9H), 13C-NMR ($CDCl_3$) 34 7, 109 7, 118 3, 123 1, 123 7, 124 1, 126 3, 127 7, 128 4, 134 2, 136 2, 141 9, 151 8, 164 2, MS(m/e) 267(M, 15), 117(100), 115(40), **10**: m p 134-6C, 1H-NMR ($CDCl_3$) 5 02 (d, J = 6 1 Hz, 2H), 6 28 (dt J = 15 7 and 6 1 Hz, 1H), 6 72 (d, J = 15 7 Hz, 1H), 7 1-7 9 (m, 5H), 13C-NMR ($CDCl_3$) 47 8, 109 6, 110 3, 120 3, 124 2, 124 8, 128 2, 128 5, 131 5, 132 3, 134 7, 135 5, 147.0, 180 7, MS(m/e) 267(M, 16), 176(30), 117(100), 115(50), 91(17) Anal Calcd for $C_{16}H_{13}NOS$ C, 71 88, H, 4 90, N, 5 24, S, 11 99 Found C, 71 27, H, 4 95, N, 5 27, S, 11 28

2-(Cinnamylthio)benzothiazole, **11** (Run 3) This compound was obtained as for **8** A solid, probably bis(2-mercaptobenzothiazole-2-thiolate)bis(triphenylphosphine)palladium(II), m p 230-1C (Lit [18] m p 248C) was separated by digestion in dichloromethane and filtered off The solution was evaporated to afford **11** , m p 60-1 C (diethyl ether-pentane), 1H-NMR ($CDCl_3$) 4 11 (d, J = 6 8 Hz, 2H), 6 27 (dt, J = 15 7 and 6 8 Hz, 1H), 6 64 (d, J = 15 7 Hz, 1H), 7 10-7 90 (m, 9H), 13C-NMR ($CDCl_3$) 35 9, 120 8, 121 4, 123 4, 124 1, 125 9, 126 3, 127 7, 128 4, 134 1, 135 2, 136 2, 153 1, 166 0, MS(m/e) 283(M, 40), 117(100), 115(45), 91(14) Anal Calcd for $C_{16}H_{13}NS_2$ C, 67 81, H, 4 62, N, 4 92, S, 22 63 Found C, 67 07, H, 4 65, N, 5 28, S, 23 11

2-(Cinnamylthio)benzimidazole, **12** (Run 4) This compound was obtained as for **8** No precipitate was observed The solvent was evaporated and the residue digested in chloroform to give insoluble

thiobenzimidazolone, **5**, (0 275 g, 26% recovery) Compound **12** precipitated by concentrating the chloroform solution and had m p 176-7C (Lit [19] m p. 172-3C); 1H-NMR (d_6-acetone) 4.13 (d, J = 5 9 Hz, 2H), 6 41 (dt, J = 14 8 and 5.9 Hz, 1H), 6.70 (d, J = 14 8 Hz, 1H), 7 00-7 52 (m, 9H), 11 37 (broad s, 1H), 13C-NMR (d_6-acetone) 36.3, 114 9, 123 4, 125.2, 127 3, 128 7, 129.5, 137 9, 150.9; MS(m/e) 266(M, 27), 175(23), 117(100), 115(57) The residual chloroform solution was evaporated and the residue was chromatographed More **12** and minor amounts of a N-cinnamyl derivative (δ = 5 03) were detected

1-Cinnamyl-2-(cinnamylthio)benzimidazole, **13**, and 1,3-dicinnamylthiobenzimidazolone, **14** (Run 5) These compounds were prepared as for **12** No precipitate was observed in the reaction medium. The reaction solvent was evaporated and the residue was chromatographed through a silica-gel column to afford **14**· m p 162-3C (ethyl acetate-hexane), 1H-NMR ($CDCl_3$) 5 19 (d, J = 5 2 Hz, 4H), 6.32 (dt, J = 13 9 and 5.2 Hz, 2H), 6 65 (d, J = 13 9 Hz, 2H), 7 10-7 30 (m, 14H), 13C-NMR ($CDCl_3$) 47 0, 109,4, 122 5, 122 9, 126 4, 127 8, 128 4, 131 9, 133 5, 136 0, 169 0; MS(m/e) 382(M, 11), 117(94), 115(100). Anal Calcd for $C_{25}H_{22}N_2S$ C, 78 49, H, 5 80, N, 7 32, S, 8 38 Found C, 78 41; H, 5 75, N, 7 31, S, 8 07, **13** m p 90-1C (ethyl acetate-hexane), 1H-NMR ($CDCl_3$) 4 22 (d, J = 7 3 Hz, 2H), 4 87 (d, J = 6 1 Hz, 2H), 6 21 (dt, J = 15 9 and 6 1 Hz, 1H), 6 38 (dt, J = 15 9 and 7 3 Hz, 1H), 6 46 (d, J = 15 9 Hz, 1H), 6 63 (d, J = 15 9 Hz, 1H), 7 10-7 30 (m, 13H), 7 66 (d, J = 9 1 Hz, 1H), 13C-NMR ($CDCl_3$) 35 5, 45 8, 109 0, 118 3, 121 9, 122 0, 122 6, 124 0, 126 3, 127 6, 127 9, 128 4, 132 9, 133 7, 135 7, 135 9, 136 3, 143 5, 151 0, MS(m/e) 382(M, 11), 291(32), 117(92), 115(100), 91(50) Anal. calcd for $C_{25}H_{22}N_2S$ C, 78 50, H, 5 80, N, 7.32 Found C, 77 51, H, 5 79, N, 7 00; and a Pd complex of unknown structure

1-Cinnamyl-1,2,4-triazole, **15a** (Run 6) A solution of $Pd(acac)_2$ (0 22 g, 0 7 mmole) and triphenylphosphine (0 75 g, 2 8 mmole) in anhydrous THF (20 mL) was added under stirring and in an argon atmosphere over a solution of 1,2,4-triazole (1 0 g, 14 2 mmole) in anhydrous THF (20 mL). To this solution cinnamyl ethyl carbonate, **1a**, (5 85 g, 28 4 mmole) in anhydrous THF (10 mL) was finally added The mixture was refluxed for 15 h The solvent was evaporated and the residue was chromatographed through a silica-gel column to afford a mixture of triphenylphosphine and 3,3-dicinnamylpentane-2,4-dione and a mixture of **15a** and triphenylphosphine oxide (2 8 g) To the last mixture containing **15a**, a solution of 75 8% wet naphthalenedisulfonic acid (2 88 g, 7 6 mmole) in ethanol was added After keeping in the refrigerator a salt (2 2 g, 47 %) of stoichiometry 1,5-NDSA **15a** = 1 2 precipitated and it was filtered off m p 230-3C, 1H-NMR (d_6-DMSO) 5 12 (d, J = 5.0 Hz, 4H), 6 44 (dt, J = 16 0 and 5 0 Hz, 2H), 6 75 (d, J = 16 0 Hz, 2H), 7 28-7 45 (m, 12H), 8 00 (d, J = 7 5 Hz, 2H), 8 50 (s, 2H), 8 91 (d, J = 8 5 Hz, 2H), 9 25 (s, 2H) The salt was partitioned with aqueous sodium hydrogen carbonate and dichloromethane The organic layer was washed with water, dried and evaporated to afford **15a**, m p 58-60C, 1H-NMR ($CDCl_3$) 5 03 (d, J = 5 0 Hz, 2H), 6 39 (dt, J = 16 0 and 5 0 Hz, 1H), 6 61 (d, J = 16 0 Hz, 1H), 7 25-7 50 (m, 5H), 8.03 (s, 1H), 8 19 (s, 1H) Anal Calcd for $C_{11}H_{11}N_3$ C, 71 33, H, 5 98, N, 22 68 Found C, 71 56, H, 6 03, N, 22 67

1-(2-Cyclohexen-1-yl)-1,2,4-triazole, **15b** (Run 8) This compound was prepared as for **15a** The reaction solvent was evaporated and a solution of 78 5% wet naphthalene-1,5-disulfonic acid (1 91 g, 5 0 mmole) in ethanol (10 mL) was directly added to the residue The salt (1 65 g, 78%) (1,5-NDSA **15b** = 1 2) precipitated and was filtered off m p 182-4C; 1H-NMR (d_6-DMSO) 1 47-2 25 (m, 12H), 4 96-5 28 (m, 2H), 5 62-6 25 (m, 4H), 7 34 (d, J = 7 0 Hz, 1H), 7 46 (d, J = 7 0 Hz, 1H), 7 93 (dd, J = 7 0 and 1 3 Hz, 2H), 8 41 (s, 2H), 8 87 (dd, J = 7 0 and 1 3 Hz, 2H), 9 06 (s, 2H) Anal Calcd for $C_{26}H_{30}N_6O_6S_2$ C, 53 23, H, 5 15, N, 14 32 Found C, 53 07, H, 5 09, N, 14 17 Free **15b**, oil, 1H-NMR ($CDCl_3$) 1 47-2 31 (m, 6H), 4 80-5 90 (m, 1H), 5 70-5 91 (m, 1H), 6.06-6 28 (m, 1H), 7 94 (s, 1H), 8 12 (s, 1H)

1-Allyl-1,2,4-triazole, **15c** (Run 9) This compound was prepared as for **15b** Naphthalene-1,5-disulfonate of **15c** (1 2 stoichiometry)· m p 189-192C, 1H-NMR (d_6-DMSO) 4 91 (d, J = 5 0 Hz, 4H), 5 09-5 37 (m, 4H), 5 81-6 44 (m, 2H), 7 42 (dd, J = 7.0 and 7 0Hz, 2H), 7 96 (dd, J = 7 0 and 1 3 Hz, 2H), 8 37 (s, 2H), 8 87 (dd, J = 7 0 and 1 3 Hz, 2H), 9 03 (s, 2H) Anal Calcd for $C_{20}H_{22}N_6O_6S_2$ C, 47 42, H, 4 38, N, 16 59 Found C, 47 20, H, 4 29, N, 16 25 Free **15c**, oil, 1H-NMR ($CDCl_3$) 4 69 (d, J = 5 0 Hz, 2H), 5 03-5 34 (m, 2H), 5 69-6 17 (m, 1H), 7 84 (s, 1H), 7 97 (s, 1H)

1-(2-Methylallyl)-1,2,4-triazole, **15d** (Run 10) This compound was prepared as for **15b** Naphthalene-1,5-disulfonate of **15d** (1 2 stoichiometry) 199-202C, 1H-NMR (d_6-DMSO) 1 66 (s, 6H), 4 76(s, 2H), 4 84 (s, 4H), 5 00 (s, 2H), 7 43 (dd J = 7 0 and 7.0, Hz, 2H), 7 97 (dd, J = 7 0 and 1.3 Hz, 2H), 8 37 (s, 2H), 8 87 (dd J = 7 0 and 1.3 Hz, 2H), 9 03 (s, 2H) Anal Calcd for $C_{22}H_{26}N_6O_6S_2$ C, 49 43, H, 4 90, N, 15 72 Found C, 49 11, H, 4 83, N, 15 61 Free **15d**, oil, 1H-NMR ($CDCl_3$) 1.59 (s, 3H), 4 61 (s, 2H), 4 75 (s, 1H), 4 91 (s, 1H), 7 84 (s, 1H), 7 97 (s, 1H)

3(5)-(Cinnamylthio)-1,2,4-triazole, **16**, 1-cinnamyl-5-(cinnamylthio)-1,2,4-triazole, **17**, and 1-cinnamyl-3-(cinnamylthio)-1,2,4-triazole, **18** (Run 11) Compounds **16-18** were prepared as for **15b**. The reaction solvent was evaporated and the residue chromatographed through a column of silica gel to afford, in elution order **17**: m.p. 53-5C (diethyl ether), 1H-NMR ($CDCl_3$) 4 00 (d, J = 7 3 Hz, 2H), , 4.83 (d, J = 6 1 Hz, 2H), 6 19 (dt, J = 15 9 and 6 1 Hz, 1H), 6.26 (dt, J = 15 9 and 7 3 Hz, 1H), 6 51 (two d, J = 15 9 Hz, 2H), 7 20-7 38 (m, 10H), 7 93 (s, 1H), 13C-NMR ($CDCl_3$) 36.3, 50.6, 121 9, 123 4, 126.3, 126.5, 127 7, 128 0, 128 4, 133 8, 134 1, 135 5, 136 0, 150 9, 151 4 Anal Calcd for $C_{20}H_{19}N_3S$. C, 72 04, H, 5 74, N, 12 60 Found C, 72 09; H,5 71, N, 12.60, **18** m p 83-5C (diethyl ether), 1H-NMR ($CDCl_3$) 3 90 (d, J = 7 3 Hz, 2H), 4 84 (d, J = 6 7 Hz, 2H), 6 26 (dt, J = 15 9 and 6.7 Hz, 1H), 6.29 (dt, J = 15 9 and 7.3 Hz, 1H), 6 53 (d, J = 15 9 Hz, 1H), 6 59 (d, J = 15 9 Hz, 1H), 7 14-7 32 (m 10H), 8 03 (s, 1H), 13C-NMR ($CDCl_3$). 34 5, 51 7, 121 6, 124 7, 126 2, 126 5, 127 3, 128 2, 128 3, 128.5, 132 8, 134 9, 135.3, 136 4, 143 5, 160 1 Anal. Calcd for $C_{20}H_{19}N_3S$ C, 72 04, H, 5 74, N, 12.60 Found C, 72.00, H, 5 72, N, 12 69, **16** m p 124-5C, IR(KBr) 3114-2715 (broad) cm^{-1}, 1H-NMR (d_4-Methanol) 3 88 (d, J = 6 3 Hz, 2H), 6 23 (dt, J = 16.3 and 6 3 Hz, 1H), 6 52 (d, J = 16 3 Hz, 1H), 7 13-7 38 (m, 5H), 8 25 (s, 1H), 13C-NMR (d_6-DMSO). 34 0, 125 2, 126 1, 127 5, 128.4, 132.3, 136 2. Anal Calcd. for $C_{11}H_{11}N_3S$ C, 60 80, H, 5 10, N, 19.34. Found C, 60 72, H, 5 03, N, 19 25

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