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Synthesis and Characterization of Novel *N*-Ferrocenesulfonyl Benzimidazole

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Abstract: A new series of ferrocenesulfonyl benzimidazle has been synthesized and characterized by ^{1}H NMR, FT-IR and elemental analysis. They are expected to have special bio-activity.

Keywords: *N*-Ferrocenesulfonyl benzimidazole, synthesis, characterization.

Ferrocene derivatives are important not only in theory but also in practical application. The chemistry of ferrocene derivatives has been the most hot research area since its discovery in 1951. In 1975, Edwards synthesized ferrocenyl penicillin and ferrocenyl cephalosporin, and found they had good antimicrobial activity[1]. Through reaction of aminoferrocene with isosulfocyanic ester, Yuan, Y. F. and co-workers prepared in 1997 a series of *N*-ferrocenyl-*N*'-aryl substituted thioureas and predicted they had special activity on plant growth[2]. In 1998, ferrocenesulfonamide was converted to *N*-(ferrocenesulfonyl)ureas and *N*-(ferrocenesulfonyl)carbomates by Becenyei and Parkanyi *via* reacting ferrocenesulfonamide with the corresponding isocyanates or chloroformic acid esters[3].

Sulfonamide derivatives represent one of the most active classes of compounds possessing a wide spectrum of biological activity. And, benzimidazole derivatives exhibit special biological activity such as antibacterial, antitumor and antiviral activities. In view of these facts, it is urgent and interesting to introduce ferrocenesulfonyl moiety into benzimidazole to obtain compounds possessing potential biological activity.

In this paper, we first reported the synthesis of a new series of *N*-ferrocenesulfonyl benzimidazole and they are expected to have special biological activity.

Melting points were determined with a XT-4 micro melting point apparatus and the thermometer was uncorrected. IR spectra were recorded on a EQUINOX-55 spectrometer in KBr tablet. ^{1}H NMR spectra were measured on a Bruker-400 spectometer using TMS as internal standard and $CDCl_3$ as solvent. Elemental analysis was performed on a PE-2400 elemental analyzer.

Ferrocenesulfonyl chloride was synthesized according to D. W. Slocum's method[4]. 2-Substituted benzimidazole(**2a-e**) was prepared according to D. G. Bapat's method[5] and was identified by its melting point[6,7].

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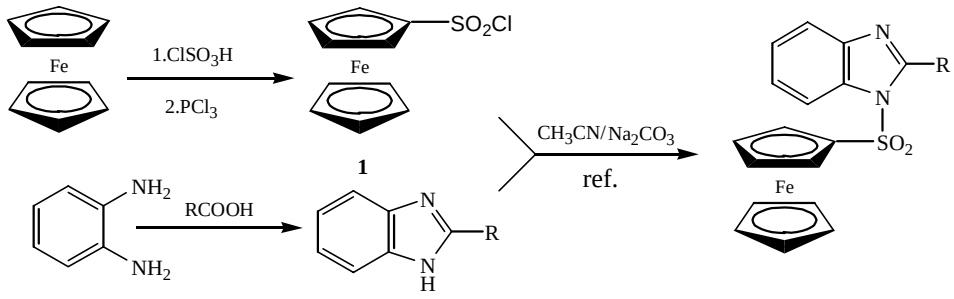
Scheme 1



a: R = H b: R=CH_3 c: R= n-C_4H_9 d: R= C_6H_5 e: R= 4-ClC_6H_4

Table 1 Physical data and elemental analysis of compounds **3a-e**

compd.	mp. (℃)	reaction time(h)	yields (%)	elemental analysis (found) C	H	N	S
3a	197-199	20	56	55.75(55.72)	3.85 (3.78)	7.65 (7.69)	8.76 (8.96)
3b	193-195	20	54	56.86 (56.92)	4.24 (4.21)	7.36 (7.24)	8.43 (8.43)
3c	166-168	36	45	59.72 (59.69)	5.25 (5.17)	6.63 (6.57)	7.59 (7.75)
3d	195-197	48	42	62.45 (62.39)	4.10 (4.05)	6.33 (6.26)	7.25 (7.42)
3e	203-205	48	40	57.94 (57.86)	3.59 (3.60)	5.88 (5.55)	6.72 (6.63)

Table 2 IR and ^{1}H NMR spectra data of compounds **3a-e**

Compd.	IR(cm^{-1})	^{1}H NMR ($CDCl_3$, δ,ppm)
3a	3107,1642,1604,1499,1366, 1252,1134,1028, 492	4.462~4.816(t, 9H, Fc-H), 7.375~7.824(m, 4H, Ar-H), 8.296 (s, 1H, CH)
3b	3092,1638,1540,1370,1242, 1143,1050, 487	2.791(s,3H,CH_3), 4.430~4.787 (t,9H,Fc-H), 7.314~7.919(m, 4H, Ar-H)
3c	3096,2953,2863,1641,1538, 1369,1254, 1137,1042,493	0.990 (m, 3H, CH_3), 1.509(m, 2H, CH_2), 1.887 (m, 2H, CH_2), 3.129(m, 2H, CH_2), 4.429~4.777(t, 9H, Fc-H),7..314~7.924 (m, 4H, Ar-H)
3d	3104,1646,1530,1374,1204, 1146,1019, 490	4.312~4.393 (d, 9H, Fc-H), 7.425~8.103 (m, 9H, Ar-H)
3e	3083,1648,1522,1378,1203, 1146,1008, 497	4.314~4.395 (d, 9H, Fc-H), 7.405~8.082 (m, 8H, Ar-H)

Under nitrogen atmosphere, 1.42 g (5 mmol) ferrocenesulfonyl chloride and 8 mmol 2-substituted benzimidazole were dissolved in 30 mL dried acetonitrile in a 50mL

three-necked round-bottomed flask fitted with a condenser and a mechanical stirrer. To this solution anhydrous sodium carbonate was added. The reaction mixture was then stirred and refluxed. When the reaction was over, the reaction mixture was filtered and the filtrate evaporated to dryness under vacuum. The residue obtained was then purified by column chromatography(100-200 mesh silica gel, $CHCl_3$, Rf = 0.6) and recrystalized from $CHCl_3$ to give the pure compounds **3a-e** as yellow crystals. The traditional system for this reaction is $(C_2H_5)_2O/C_5H_5N$. We have tried this reaction in 3 systems: $(C_2H_5)_2O/C_5H_5N$, C_6H_6/C_5H_5N and CH_3CN/Na_2CO_3. No reaction taken place in the first system, perhaps due to the low reaction temperature. In the second system, the reaction is slow. The last system gives best results ever tried.

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References

1. E. I. Edwards, R. Epton, G. Marr, *J. Organomet. Chem*., **1975**, *85*, 23.
2. Y. F. Yuan, B. Wang, J. T. Wang, *Hua Xue Tong Bao* ,1997, *12*, 43.
3. G. Besenyei, L. Parkanyi, *J. Organomet. Chem*., **1998**, *563*, 81.
4. D. W. Slocum, W. Achermann, *Synth. React. Inorg. Met.-Org. Chem*., **1982**, *12* (4), 397
5. D. G. Bapat, M. V. Shirsat, *Indian J. Chem*., **1965**, *3*, 81.
6. W. O. Pool, H. J. Harward, A. W. Ralston, *J. Am. Chem. Soc*., **1937**, *59*, 178.
7. V. I. Cohen, *J. Heterocycl. Chem*.,**1979**, *16*, 13.

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