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MICROWAVE ASSISTED SYNTHESIS OF 4-(SUBSTITUTED FLUORO-PHENYL)-SUBSTITUTED-6H-1-THIA-5,7,8,9A-TETRAAZA-CYCLOPENTA[E]AZULENES DERIVATIVES.

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ABSTRACT:

We have synthesized 4-(substituted fluoro-phenyl)-substituted-6*H*-1-thia-5, 7, 8, 9a-tetraaza-cyclopenta[*e*]azulene derivatives using (2-Amino-5-substituted-thiophen-3-yl)-(substituted-fluoro-phenyl)-methanone and (1, 3-Dioxo-1,3-dihydro-isoindol-2-yl)-acetic acid with the help of microwave irradiation technique. Initially equimolar solution of (2-Amino-5-substituted-thiophen-3-yl)-(substituted-fluoro-phenyl)-methanone and (1,3-Dioxo-1,3-dihydro-isoindol-2-yl)-acetic acid in DCM were reacted in presence of amide coupling agent T3P and triethyl amine catalyst in microwave at RT for 5-10 min. to form intermediate 2-(1,3-Dioxo-1,3-dihydro-isoindol-2-yl)-N-[3-(fluoro-benzoyl)-5-substituted-thiophen-2-yl]-acetamide with 50-60 % of isolated yield. Compound2-(1,3-Dioxo-1,3-dihydro-isoindol-2-yl)-N-[3-(fluoro-benzoyl)-5-substituted-thiophen-2-yl]-acetamide were irradiated with excess of hydrazine hydrate and DMF in microwave at 80-90 ^{O}C for 50-60 min to afford compounds 7-substituted-5-phenyl-1,3-dihydro-thieno[2,3-e][1,4]diazepin-2-one with isolated yield.

Keywords: Coupling agent T3P, DMF, Microwave, Acetamide, Hydrazine Hydrate, substituted fluoro-phenyl.

1. INTRODUCTION

Heterocyclic compounds are the most important and maintain a vigorous field in organic chemistry. They play a significant role in the drug design and synthesis process. Ahmet Cansiz and Co. worker[1]reported synthesized of 6-Phenyl-3-(4-pyridyl)-1,2,4-triazolo-[3,4-b][1,3,4]thiadiazole by multicomponent reaction starting from ionized. various thiazole derivatives synthesis and their biological activities are reported for 1,2,4-triazolo[3,4-b]-1,3,4-thiadiazoles and its derivatives containing pyridine moiety. Sadaf J. Gilani et.al [2] reported the anti-microbial activity of synthesized 6-substituted-1, 2, 4-triazolo-[3, 4-*b*]-1, 3, 4-thiadiazole derivatives, by comparison, most of the compound displayed excellent antimicrobial activities against tested microorganism. Ram J. Singh and co-workers [3] reported microbial activity of newly synthesized derivatives of 3-pyridyl-6-aryl-s-triazolo [3, 4-b]-[1, 3, 4]-thiadiazoles. Screening result shows that 1,2,4-triazole moiety at 1 or 2 position exhibits sensible antibacterial activity against tested microorganisms. Bhat K. S. and workers[4]synthesized fluorinated3,6-diaryl-[1,2,4]triazolo [3,4-b][1,3,4] thiadiazole derivatives demonstrating action against human breast cancer, human osteosarcoma and human myeloid leukaemia has reported. Among these, the compound possesses higher anti-proliferative activity. Chai B.

etal[5]were synthesized 6-aryl-3-{(4-substituted phenoxy) methyl}-1,2,4-triazolo[3,4-b]-1,3,4-thiadiazoles against pathogenic strains and fungi.
Prasad D. J. [6]synthesized 3,6-disubstituted-1,2,4-triazolo[3,4-b]-1,3,4-thiadiazoles derivatives showing anti-inflammatory and analgesic activity. Reported derivatives were found potent and active against tested pathogens by comparing standard drugs. Yassin F. A. and Seleim A. F.[7] studied anti-inflammatory and antimicrobial activities of 1,2,4-triazolo[3,4-b][1,3,4] thiadiazoles containing trichloro phenyl compounds against different strains. Reported derivatives showed excellent anti-inflammatory and anti-microbial activity. Sahin D. and worker[8] reported the synthesis of [1,2,4]-triazolo-[3,4-b][1,3,4]thiadiazole compounds showing good microbial activities against tested microorganisms with standard drugs.
Abdulla I. Q.et al[9] synthesized and evaluated bridgehead nitrogen heterocyclic system 3,6-disubstituted-1,2,4-triazolo-[3,4-b]-1,3,4-thiadiazoles of biphenyl-4-yloxy acetic acid and ibuprofen for antibacterial, antifungal, anti-inflammatory, analgesic, ulcerogenic, and lipid peroxidation activities. The authors found that the compounds exhibit moderate microbial activity compared with standard drugs flurbiprofen and ibuprofen. Derivatives of 3-diphenylmethyl-6-substituted-1, 2 ,4-triazolo[3,4-b]-1,3,4-thiadiazole were synthesized, biologically screened for anti-inflammatory activity by Ansari K. F.et al[10-11]. Designed derivatives, with having a chloro substituent exhibited more potent activity when comparable to standard drug.
Desabattina V.N.K. and co-workers [12] synthesized a series of di-substituted 1, 2, 4-triazolo-1, 3, 4-thiadiazole derivatives and screened its antimicrobial activity [13-16]. All the compounds showed efficient inhibition towards the bacteria and fungi strains under tested.

Experimental

General Procedure for Synthesis of 2-(1, 3-Dioxo-1, 3-Dihydro-Isoindol-2-Yl)-N-[3-(Fluoro-Benzoyl)-5-Substituted-Thiophen-2-Yl]-Acetamide 3:

An equimolar amount of (2-Amino-5-substituted-thiophen-3-yl)-(substituted-fluoro-phenyl)-methanone **1 and** (1,3-Dioxo-1,3-dihydro-isoindol-2-yl)-acetic acid**2**were reacted in presence of amide coupling agent T3P (3%), catalytic triethyl amine and 10 volume of dry DCM solvent in microwave tube at RT for 5-10 min. The progress of the reaction was monitored on TLC (5% MeOH: DCM) and after completion reaction mass was quenched with ice-cold water and extracted with DCM two times and the organic layer dried over sodium sulphate and concentrated to afford compounds 2-(1,3-Dioxo-1,3-dihydro-isoindol-2-yl)-N-[3-(fluoro-benzoyl)-5-substituted-thiophen-2-yl]-acetamide **3**with 50-60 % of isolated yield.

General Procedure For Synthesis of 7-Substituted-5-Phenyl-1, 3-Dihydro-Thieno [2, 3-E] [1, 4]Diazepin-2-One 4:

1 equivalent of 2-(1,3-Dioxo-1,3-dihydro-isoindol-2-yl)-N-[3-(fluoro-benzoyl)-5-substituted-thiophen-2-yl]-acetamide **3** were irradiated with 2 equivalent hydrazine hydrate and 10 volume of DMF in microwave tube at 80-90 ^{O}C for 50-60 min. The progress of the reaction was monitored on TLC (30% EA: Hex) and after completion reaction mass was quenched with ice-cold water and extracted with EtOAC two times and the organic layer dried over sodium sulphate and concentrate to afford compounds 7-substituted-5-phenyl-1,3-dihydro-thieno[2,3-e][1,4]diazepin-2-one 4 with 74-80 % of isolated yield.

General Procedure For Synthesis Of 7-Substituted-5-Phenyl-1,3-Dihydro-Thieno[2,3-E][1,4]Diazepine-2-Thione 5:

7-substituted-5-phenyl-1,3-dihydro-thieno[2,3-e][1,4]diazepin-2-one 4 (1 equiv.) were irradiated with Lawessons reagent (2 equiv.) And 10 volume of DCM in microwave tube at 40-45 ^{O}C for 30-40 min. The progress of the reaction was monitored on TLC (10% EA: Hex)

and after completion reaction mass was quenched with ice-cold water and neutralized with saturated sodium bicarbonate solution resultant solids were filtered out and washed with excess water to afford compounds 7-substituted-5-phenyl-1,3-dihydro-thieno[2,3-e][1,4]diazepine-2-thione **5**with 74-80 % of isolated yield.

General procedure for synthesis of 4-(substituted fluoro-phenyl)-substituted-6*H*-1-thia-5, 7, 8, 9a-tetraaza-cyclopenta[*e*]azulene derivatives 7(a-j):

Equimolar amount of 7-substituted-5-phenyl-1, 3-dihydro-thieno [2, 3-e] [1,4]diazepine-2-thione 5 and substituted acid hydrazide 6 were reacted in the presence of $POCl_3$ in microwave tube at 90-95 ^{O}C for 50-60 min. The progress of the reaction was monitored on TLC (20% EA: Hex) and after completion reaction mass was quenched with ice-cold water and neutralized with saturated sodium bicarbonate solution resultant solids were filtered out and washed with excess water. Obtained crude was purified by flash chromatography (Eluting with 10 EA: Hex.) to afford compounds 4-(substituted fluoro-phenyl)-substituted-6*H*-1-thia-5, 7, 8, 9a-tetraaza-cyclopenta[*e*]-azulene derivatives 7(a-j) with 80-89 % of isolated yield.

Spectral Data

4-(2-Fluoro-phenyl)-2, 9-dimethyl-6H-1-thia-5,7,8,9a-tetraaza-cyclopenta[e]azulene (7a).

Melting Point: 185^{0}C. ^{1}H NMR (400 MHz, DMSO, δ ppm): 2.31 (s, 3H, -CH_3), 2.70 (s, 3H, -CH_3), 4.60 (s, 1H, -CH_2), 6.51 (s, 1H, Ar-H), 7.12 (d, 1H, Ar-H), 7.20 (dd, 1H, Ar-H), 7.43 (dd, 1H, Ar-H), 7.57 (d, 1H, Ar-H). 16.80. ^{13}C NMR (400 MHz, DMSO, δ ppm): 23.61, 42.28, 115.60, 123.30, 124.24, 124.03, 125.21, 127.12, 130.36, 132.56, 139.55, 160.02, 160.88, 162.62, 164. 66.

9-Ethyl-4-(2-Fluoro-Phenyl)-2-Methyl-6H-1-Thia-5,7,8,9a-Tetraaza cyclopenta[e]azulene(7b).

Melting Point: 201^{0}C. ^{1}H NMR (400 MHz, DMSO, δ ppm): 1.48 (t, 3H, -CH_3), 2.81 (s, 3H, -CH_3), 2.99 (q, 2H, -CH_2), 4.41 (s, 1H, -CH_2), 6.56 (s, 1H, Ar-H), 7.23 (d, 1H, Ar-H), 7.20 (dd, 1H, Ar-H), 7.52 (dd, 1H, Ar-H), 7.54 (d, 1H, Ar-H). ^{13}C NMR (400 MHz, DMSO, δ ppm): 18.10, 17.78, 20.32, 42.18, 115.60, 123.34, 124.56, 124.84, 125.62, 127.00, 130.06, 132.65, 139.50, 160.23, 160.95, 162.62, 164.60.

4-(2-Fluoro-Phenyl)-2-Methyl-9-Phenyl-6H-1-Thia-5,7,8,9a-Tetraaza Cyclopenta[E]Azulene(7c).

Melting Point: 228^{0}C. ^{1}H NMR 2.15 (s, 3H, -CH_3), 4.02 (s, 1H, -CH_2), 6.56 (s, 1H, Ar-H), 7.14 (d, 1H, Ar-H), 7.24 (dd, 1H, Ar-H), 7.40 (dd, 1H, Ar-H), 7.51 (d, 1H, Ar-H), 7.60-7.98 (m, 5H, Ar-H). ^{13}C NMR (400 MHz, DMSO, δ ppm): 23.30, 42.84, 113.60, 118.20, 123.40, 124.24, 124.99, 125.21, 126.20, 127.18, 130.26, 131.23, 132.60, 139.52, 158.28, 160.00, 160.25, 162.00, 164.23.

2-Ethyl-4-(2-Fluoro-Phenyl)-9-Methyl-6H-1-Thia-5,7,8,9a-Tetraaza Cyclopenta[E]Azulene (7d).

Melting Point: 191^{0}C. ^{1}H NMR (400 MHz, DMSO, δ ppm): 1.31 (t, 3H, -CH_3), 2.71 (s, 3H, -CH_3), 2.83 (q, 2H, -CH_2), 4.91 (s, 1H, -CH_2), 6.52 (s, 1H, Ar-H), 7.03 (d, 1H, Ar-H), 7.23 (dd, 1H, Ar-H), 7.43 (dd, 1H, Ar-H), 7.54 (d, 1H, Ar-H). ^{13}C NMR (400 MHz, DMSO, δ ppm): 12.10, 15.46, 23.30, 47.06, 116.25, 122.78, 124.34, 127.10, 129.97, 131.10, 132.06, 132.96, 142.97, 149.56, 153.10, 159.07, 161.57, 162.87.

2-Ethyl-4-(2-fluoro-phenyl)-)-9-phenyl-6H-1-thia-5,7,8,9a-tetraaza cyclopenta[e]azulene(7e).
Melting Point: 230^{0}C. ^{1}H NMR (400 MHz, DMSO, δ ppm): 1.10 (t, 3H, -CH_3), 1.95 (t, 3H, -CH_3), 2.28 (q, 2H, -CH_2), 2.80 (q, 2H, -CH_2), 4.60 (s, 1H, -CH_2), 6.48 (s, 1H, Ar-H), 7.22 (d, 1H, Ar-H), 7.30 (dd, 1H, Ar-H), 7.55 (dd, 1H, Ar-H), 7.88 (d, 1H, Ar-H). ^{13}C NMR (400 MHz, DMSO, δ ppm): 14.10, 16.18, 18.20, 23.18, 42.10, 115.24, 123.30, 124.28, 124.29, 125.24, 127.63, 130.66, 132.46, 139.26, 158.14, 160.15, 162.20, 164.00.

2, 9-Diethyl-4-(2-fluoro-phenyl)-6H-1-thia-5,7,8,9a-tetraaza-cyclopenta[e]azulene(7f).
Melting Point: 270^{0}C. ^{1}H NMR (400 MHz, DMSO, δ ppm): 1.90 (t, 3H, -CH_3), 2.30 (q, 2H, -CH_2), 4.62 (s, 2H, -CH_2), 6.50 (s, 1H, Ar-H), 7.22 – 8.30 (m, 9H, Ar-H). ^{13}C NMR (400 MHz, DMSO, δ ppm): ^{13}C NMR (400 MHz, DMSO, δ ppm): 14.10, 16.18, 18.20, 23.18, 42.10, 115.24, 123.30, 124.28, 124.29, 125.24, 127.63, 130.66, 132.46, 139.26, 158.14, 160.15, 162.20, 164. 00.

4-(4-Fluoro-phenyl)-2,9-Diethyl-4-(2-fluoro-phenyl)-6H-1-thia-5,7,8,9a-tetraaza-cyclopenta[e]azulene(7g).
Melting Point: 220^{0}C. ^{1}H NMR (400 MHz, DMSO, δ ppm): 1.75 (s, 3H, -CH_3), 1.90 (s, 3H, -CH_3), 4.60 (s, 2H, -CH_2), 6.48 (s, 1H, Ar-H), 7.28 - 7.88 (m, 4H, Ar-H).

9-Ethyl-4-(4-Fluoro-Phenyl)-2,9-Diethyl-4-(2-Fluoro-Phenyl)-6H-1-Thia-5,7,8,9a-Tetraaza-Cyclopenta[E]Azulene(7h).
Melting Point: 242^{0}C. ^{1}H NMR (400 MHz, DMSO, δ ppm): 1.16 (t, 3H, -CH_3), 1.95 (t, 3H, -CH_3), 2.30 (q, 2H, -CH_2), 4.65 (s, 2H, -CH_2), 6.49 (s, 1H, Ar-H), 7.30- 7.98 (m, 4H, Ar-H).

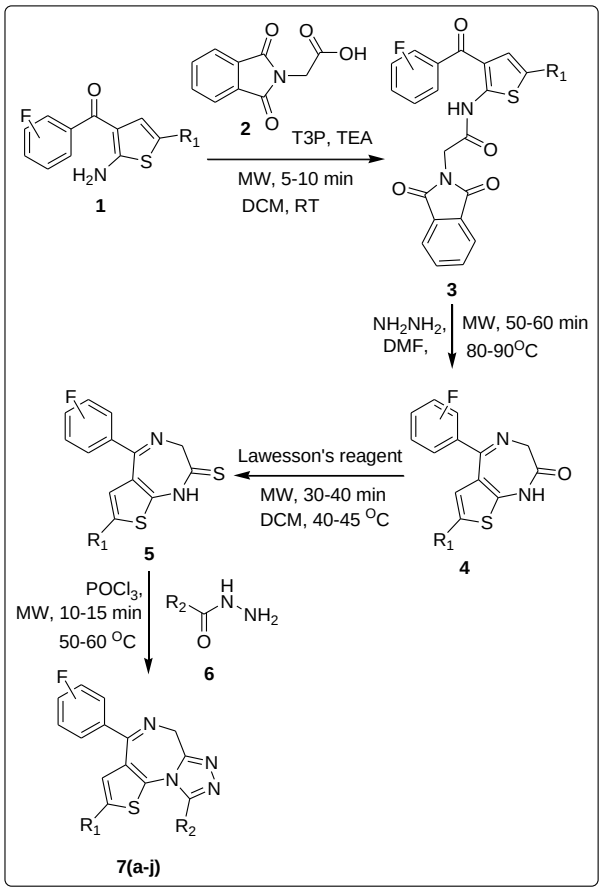
4-(4-Fluoro-Phenyl)-2-Methyl-9-Phenyl-6H-1-Thia-5,7,8,9a-Tetraaza-Cyclopenta[E]Azulene(7i).
Melting Point: 180^{0}C. ^{1}H NMR (400 MHz, DMSO, δ ppm): 1.10 (s, 3H, -CH_3), 4.58 (s, 2H, -CH_2), 6.56 (s, 1H, Ar-H), 7.38 – 8.80 (m, 9H, Ar-H).

2-ethyl-4-(4-Fluoro-phenyl)-9-methyl-6H-1-thia-5,7,8,9a-tetraaza-cyclopenta[e]azulene (7j).
Melting Point: 178^{0}C. ^{1}H NMR (400 MHz, DMSO, δ ppm): 1.15 (t, 3H, -CH_3), 1.90 (s, 3H, -CH_3), 2.30 (q, 2H, -CH_2), 4.56 (s, 2H, -CH_2), 6.52 (s, 1H, Ar-H), 7.49– 8.00 (m, 4H, Ar-H).

2. RESULT AND DISCUSSION

In the present work we have synthesize 4-(substituted fluoro-phenyl)-substituted-6*H*-1-thia-5,7,8,9a-tetraaza-cyclopenta[*e*]azulene derivatives7(a-j)using (2-Amino-5-substituted-thiophen-3-yl)-(substituted-fluoro-phenyl)-methanone 1and (1,3-Dioxo-1,3-dihydro-isoindol-2-yl)-acetic acid 2 with the help of microwave irradiation technique.
Initially equimolar solution of (2-Amino-5-substituted-thiophen-3-yl)-(substituted-fluoro-phenyl)-methanone 1 and (1,3-Dioxo-1,3-dihydro-isoindol-2-yl)-acetic acid2in DCMwere reacted in presence of amide coupling agent T3P (3%) and triethyl amine catalyst in microwave at RT for 5-10 min. to form intermediate 2-(1,3-Dioxo-1,3-dihydro-isoindol-2-yl)-N-[3-(fluoro-benzoyl)-5-substituted-thiophen-2-yl]-acetamide 3with 50-60 % of isolated yield.
Compound 2-(1,3-Dioxo-1,3-dihydro-isoindol-2-yl)-N-[3-(fluoro-benzoyl)-5-substituted-thiophen-2-yl]-acetamide 3 were irradiated with excess of hydrazine hydrate and DMF in microwave at 80-90 ^{O}C for 50-60 min to afford compounds 7-substituted-5-phenyl-1,3-

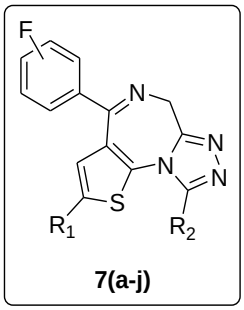
dihydro-thieno[2,3-e][1,4]diazepin-2-one **4** with 74-80 % of isolated yield.



Scheme 1:-Synthesis of 7a-j uses microwave irradiation.

Microwave irradiation of 7-substituted-5-phenyl-1,3-dihydro-thieno[2,3-e][1,4]diazepin-2-one 4 with Lawessons reagent in DCM at 40-45^{O}C afford compounds 7-substituted-5-phenyl-1,3-dihydro-thieno[2,3-e][1,4]diazepine-2-thione 5 with 74-80 % of isolated yield. Finally, equimolar solution of 7-substituted-5-phenyl-1, 3-dihydro-thieno [2,3-e][1,4]diazepine-2-thione 5 and substituted acid hydrazide 6 were reacted in the presence of $POCl_3$ in the microwave at 90-95 ^{O}C for a different time interval. After completion reaction mass was quenched with ice-cold water and neutralized with saturated sodium bicarbonate solution resultant solids were filtered out washed with excess of water. Obtained crude were purified by flash chromatography (Eluting with 10 EA: Hex.) to afford compounds 4-(substituted fluoro-phenyl)-substituted-6*H*-1-thia-5, 7, 8, 9a-tetraaza-cyclopenta[*e*]azulene derivatives 7(a-j) (Scheme 5.14) with 80-89 % of isolated yield (Table 1).

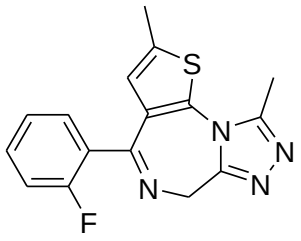
Table 1- Microwave assisted synthesis of 4-(substituted fluoro-phenyl)-substituted-6*H*-1-thia 5, 7, 8, 9a-tetraaza-cyclopenta[e]azulenes.



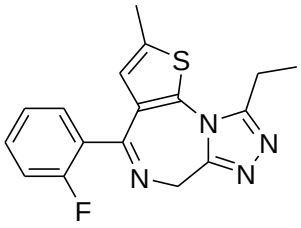
Entry	F	R_1	R_2	Time (min.)	(%)Yield[a]	M. p. ^{O}C[b]
7a	2-F	$-CH_3$	$-CH_3$	5	88	185-187
7b	2-F	$-CH_3$	$-C_2H_5$	5	86	201-205
7c	2-F	$-CH_3$	$-C_6H_5$	5	88	228-231
7d	2-F	$-C_2H_5$	$-CH_3$	10	89	191-194
7e	2-F	$-C_2H_5$	$-C_2H_5$	5	80	230-233
7f	2-F	$-C_2H_5$	$-C_6H_5$	5	89	269-271
7g	4-F	$-CH_3$	$-CH_3$	15	86	220-222
7h	4-F	$-CH_3$	$-C_2H_5$	15	85	241-244
7i	4-F	$-CH_3$	$-C_6H_5$	15	89	180
7j	4-F	$-C_2H_5$	$-CH_3$	5	82	178-180

[a]Isolated yield, [b]Melting point.

NMR methods were used to verify the compounds that were synthesised. NMR was taken at 100 MHz in DMSO and chemical shift reported in δ ppm.



7a



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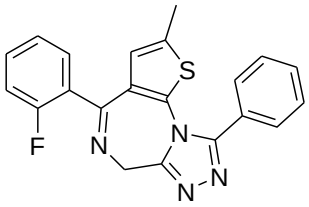

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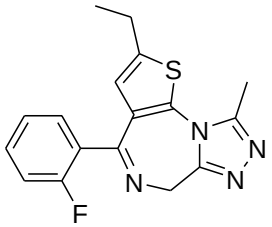

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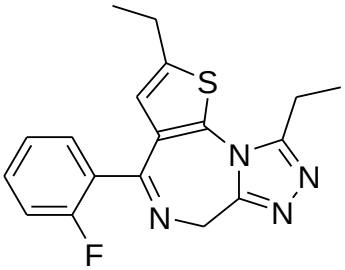

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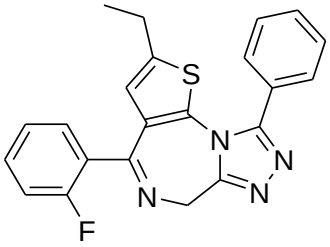

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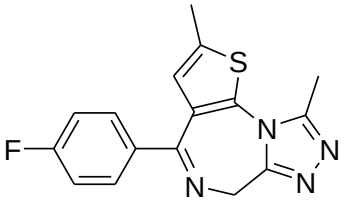

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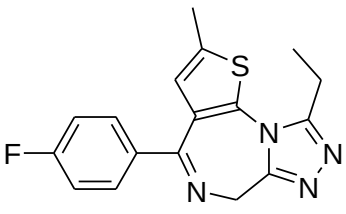

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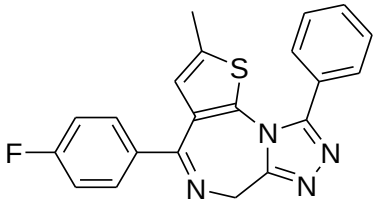

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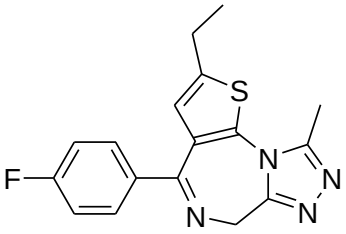

7j

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