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Design, Identification and Anti-Bacterial activity of New Tropylated Heterocycles

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ABSTRACT

This work highlights design some modern tropylated five and six substituted pyrazole and dihydropyridine hetero cyclic derivatives. These derivatives obtained by efficient one pot reaction, this one involve direct N-tropylation of substituted pyrazole and dihydropyridine derivatives in conventional alcoholic solvent under reflux condition. The chemical structures of novel designed forms proved through FT-IR, 13C-NMR, CHNS elemental analysis and anti-bacterial activity.

Keywords: *tropylateddihydropyridine, tropylatedpyrazole, 1,3,5-cycloheptatriene ring, anti-bacterial activity*

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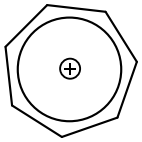
INTRODUCTION

Heterocyclic compounds involving nitrogen atom are remarkable species of compounds in the chemistry of drug and bioactive molecules [1]. The homoaromatictropylium ring carry on an essential role like benzene in aromatic chemistry, this one constitute of six pi conjugated electrons distributed on seven carbocationicring [2]



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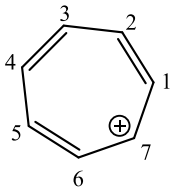

(2)

This excited cycloheptatrienemoiety is one the most important scaffold for synthesizing amino-cycloheptatriene derivatives via N-alkylation [3]metallocomplexes designing [4] participated in a different bioactive natural compounds (colchamine, colchicine) that wereempolyed to handle oncological diseases [5]functionalization of calixarenes [6]encouragingaspects of enzymes for nanomedicine and for mesomorphismfield [7].

Moreover, tropylium ring introduce antimicrobial activity reverse to several kinds of bacteria [8] against Staphylococcusaureus and Candidaalbicansstrains. [9] This fact is of obvious interest as treating of microbial eczema [10].

1,4-Dihydropyridine substituents possess vigorous biological activity [11] anti-inflammatory [12] antihypoxic and antiischemic drugs [13] calcium channel modulators of the nifedipine type [14] and use in therapeutics such as antihypertensive [15].

Pyrazolestructure construct from three carbon atoms, with two neighboring nitrogen atomsto form five-member heterocycle [16].Pyrazole is one of the most important motif might be establish many drugs, [17] complexes as ligand [18] pesticides, [19]. Pyrazole motif utilized as anti-inflammatory [20]anticancer activity [21] antifungal activity [22] anti HIV-1 activity [23] cholinesterase inhibitory activity [24] and antitubercular activity [25].

Based on the previous cited reviewer issues, the objective of this research is to design some novel N-tropylateddihydropyridine (DHP) and N-tropylatedpyrazole derivatives and in vitro testifying of gained drugs for their expected antimicrobial reactivity, we submit minimum and maximum inhibitory concentration. The synthesis of gained drugs was shown in scheme (I).

EXPERMENTAL

Apparatus measurements

Melting points of obtained compound were determined via open glass capillary tubes and all were uncorrected. TLC used to ensure purity of gained products. Purification of products achieved via recrystallization in (hexane). Infrared (IR)spectra measured via (Shimadzu Infrared Spectrophotometer Fourier Transform FTIR-8400S)utilizing KBr disc technics.13CNMR peaks recorded via (NMR broker 500 MHz). Chemical shifts are indexed in ppm and are listed corresponding to (TMS). Micro elemental analysis (CHNS) data reported via (Euro Vector EA 3000A Italy).

Organic preparation

Designing of tropyliumtetrafluoroborate (M)[26]

Into (250mL) round bottom flask with magnetic stirrer, put (6 g, 1.8 mmole) of triphenyl carbonium tetrafluoroborate and (1.9 mL, 1.8 mmole) of cycloheptatriene. Stir the mixture with minimum amount dropwise adding of acetonitrile even entire component solubility. When the solution formed wait for (5) minutes until reaction completion, after that separate the solvent out via rotatory evaporator under vacuum. The produced white precipitate was tropyliumtetra fluoroborate. The product gained isolated, washed by small portions of cold alcohol and then with cold ether so as to obtain (2.5 g) white crystals yield (78%) decompose at (198 °C)

Designing of tropylated dihydropyridine[M1-M4][27]

In a mixture of (10 mL)(5:5) distilled water: ethanol, dissolve (0.25 g, 1.4mmole)of tropyliumtetrafluroborate and equimolar ofdihydropyridine derivatives added at room temperature.

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The mixture stirred magnetically for (30) minutes and then refluxed for one hour and let to cool. The completion of reaction monitored via TLC (n-hexane 4:1 ethylacetate), neutralized with 10% solution of ammonia to PH=7 and permitted to solidify. The obtained products gathered, washed, dried and recrystallized from hexane. The yield and physical constant for gained compounds showed in table 1.

TABLE 1: some of physical properties for gained compounds (M_1-M_4)

Comp. No.	nomenclature for compounds M_1-M_4	Chemical formula of product	color	Melting point °C or decomp.	Yield %
M_1	1,1'-(1-(cyclohepta-2,4,6-trien-1-yl)-2,6-dimethyl-1,4-dihydropyridine-3,5-diyl)bis(ethan-1-one)	$C_{18}H_{21}NO_2$	yellow	138-140	60
M_2	2,2'-((1-(cyclohepta-2,4,6-trien-1-yl)-2,6-dimethyl-1,4-dihydropyridine-3,5-diyl)bis(ethan-1-yl-1-ylidene))bis(hydrazine-1-carboxamide)	$C_{20}H_{27}N_7O_2$	yellow-orange	225 decom.	63
M_3	1-(cyclohepta-2,4,6-trien-1-yl)-2,6-dimethyl-3,5-bis(1-(2-phenylhydrazineylidene)ethyl)-1,4-dihydropyridine	$C_{30}H_{33}N_5$	yellow	210-212	66
M_4	1-(cyclohepta-2,4,6-trien-1-yl)-3,5-bis(1-hydrazineylideneethyl)-2,6-dimethyl-1,4-dihydropyridine	$C_{18}H_{25}N_5$	deep yellow	232-235	64

Designing oftropylateddimethylpyrazole derivatives[M5-M8] [27]

In a mixture of (10 mL) (5:5) distilled water: ethanol, dissolve (0.25 g, 1.4mmole) of tropyliumtetrafluroborate and equimolar of dimethylpyrazole derivatives added at room temperature. The mixture stirred magnetically for (30) minutes, monitored via TLC (n-hexane 4:1 ethylacetate), neutralized with 10% solution of ammonia to PH= 7 and permitted to crystallize. The obtained products gathered, washed, dried and recrystallized from hexane. The yield and physical constant for gained compounds showedin table 2

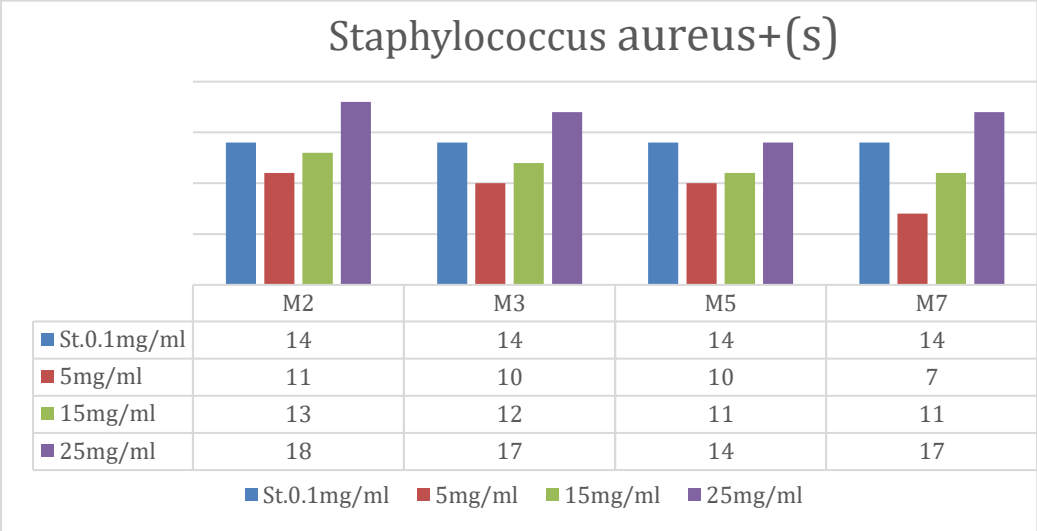
TABLE 2: some of physical properties for gained compounds (M_5-M_8)

Comp. No.	nomenclature for compounds M_5-M_8	Chemical formula of product	color	Melting point °C or decomp.	Yield %
M_5	(1-(cyclohepta-2,4,6-trien-1-yl)-2,4-dimethyl-1*H*-pyrrol-3-yl)(2-nitrophenyl)methanol	$C_{20}H_{20}N_2O_3$	yellow	123-125	65
M_6	(1-(cyclohepta-2,4,6-trien-1-yl)-2,4-dimethyl-1*H*-pyrrol-3-yl)(3-nitrophenyl)methanol	$C_{20}H_{20}N_2O_3$	yellow	2111-19	67
M_7	(1-(cyclohepta-2,4,6-trien-1-yl)-2,4-dimethyl-1*H*-pyrrol-3-yl)(4-nitrophenyl)methanol	$C_{20}H_{20}N_2O_3$	yellow	128-130	62
M_8	(4-chlorophenyl)(1-(cyclohepta-2,4,6-trien-1-yl)-2,4-dimethyl-1*H*-pyrrol-3-yl)methanol	$C_{20}H_{20}ClNO$	yellow	117-119	65

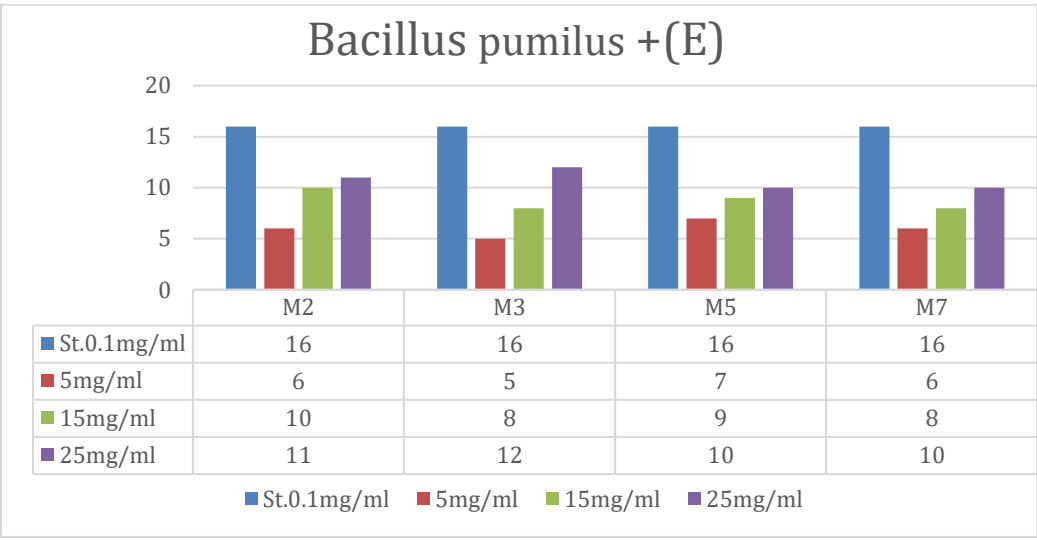
Antimicrobial activity

In our contribution, the antibacterial activity of gained compounds(M2, M3, M5, M7)were tested by three kinds of (Gram Positive) bacteria, Staphylococcus aureus, Bacillus subtilis, Bacillus pumil us by Wells method [28]. This method comprise the exposure of the zone of inhibition into the diffusion of micro- organism on agar plate. The plates incubated for 24hr, at 37C°. Neomycin concentration employed as standard drug (1 mg/mL), while the evaluated compounds (M2, M3, M5, M7) were (5, 15, 25) mg/mL. The inhibition zone diameters around every hole measured in millimeter.

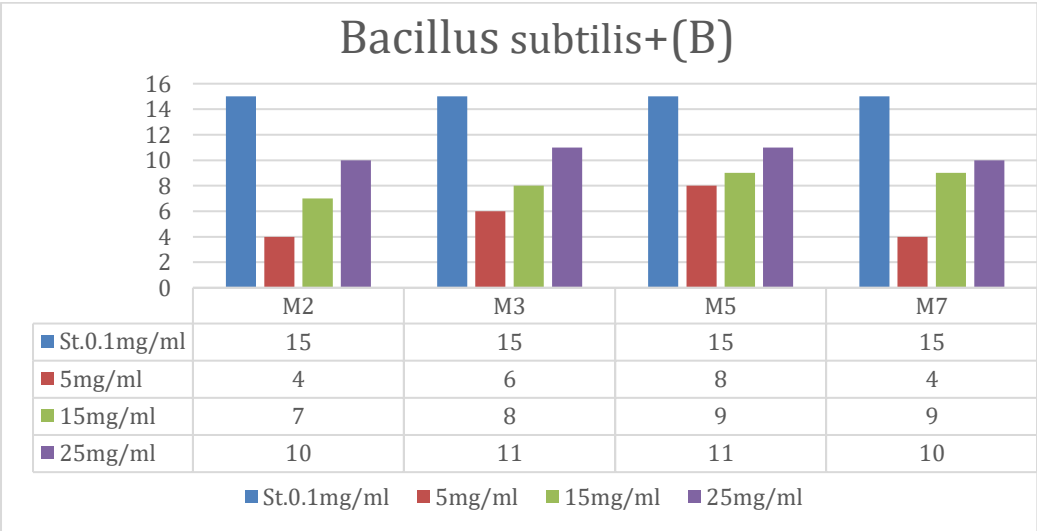
The results revealed powerful to moderate activity of all selected compounds against all kinds of tested bacteria relative the standard (Neomycin) drug at the concentration employed.



	M2	M3	M5	M7
St.0.1mg/ml	14	14	14	14
5mg/ml	11	10	10	7
15mg/ml	13	12	11	11
25mg/ml	18	17	14	17



	M2	M3	M5	M7
St.0.1mg/ml	16	16	16	16
5mg/ml	6	5	7	6
15mg/ml	10	8	9	8
25mg/ml	11	12	10	10



	M2	M3	M5	M7
St.0.1mg/ml	15	15	15	15
5mg/ml	4	6	8	4
15mg/ml	7	8	9	9
25mg/ml	10	11	11	10

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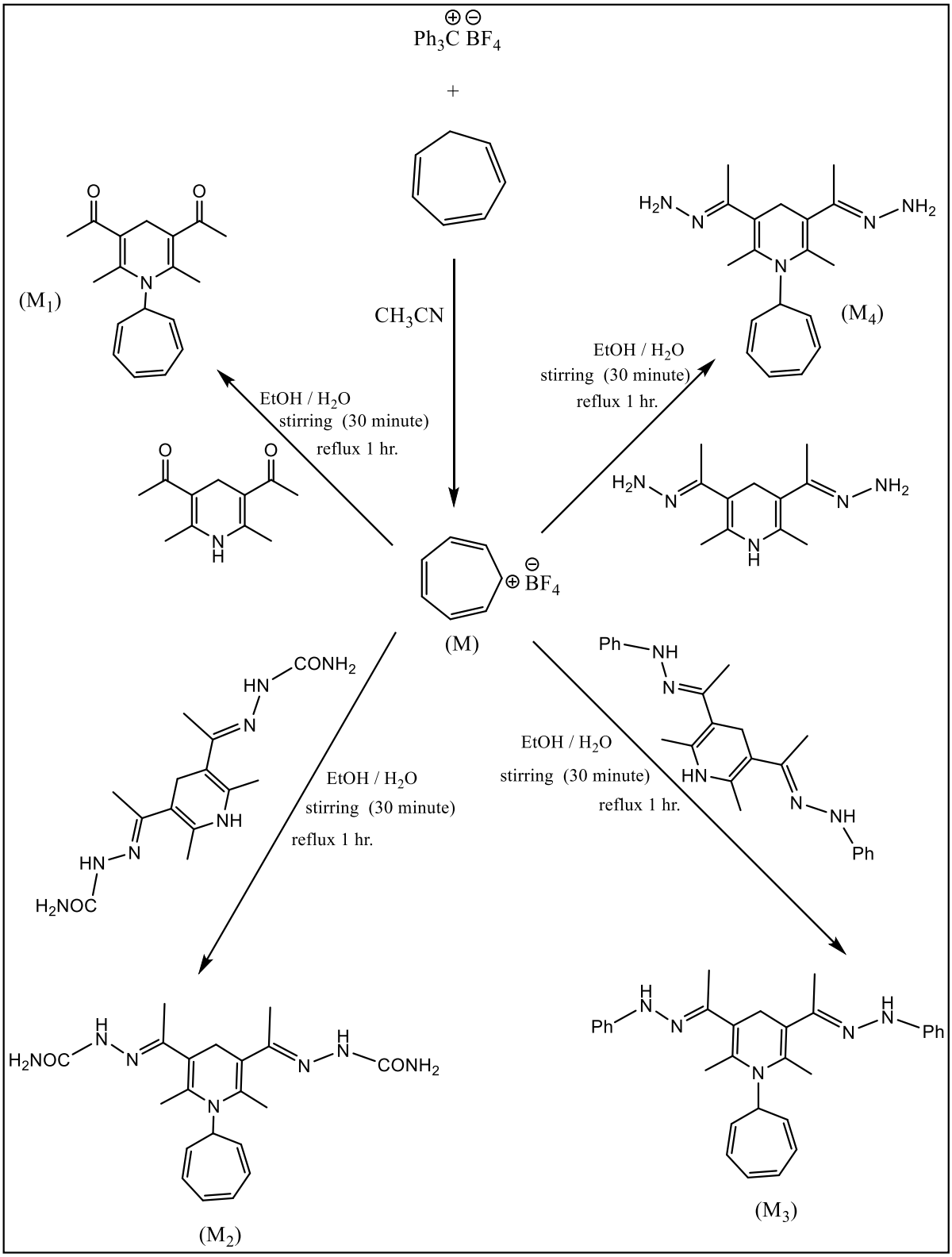

SCHEME 1: synthesis of tropylateddihydropyridine derivatives (M-M_4)

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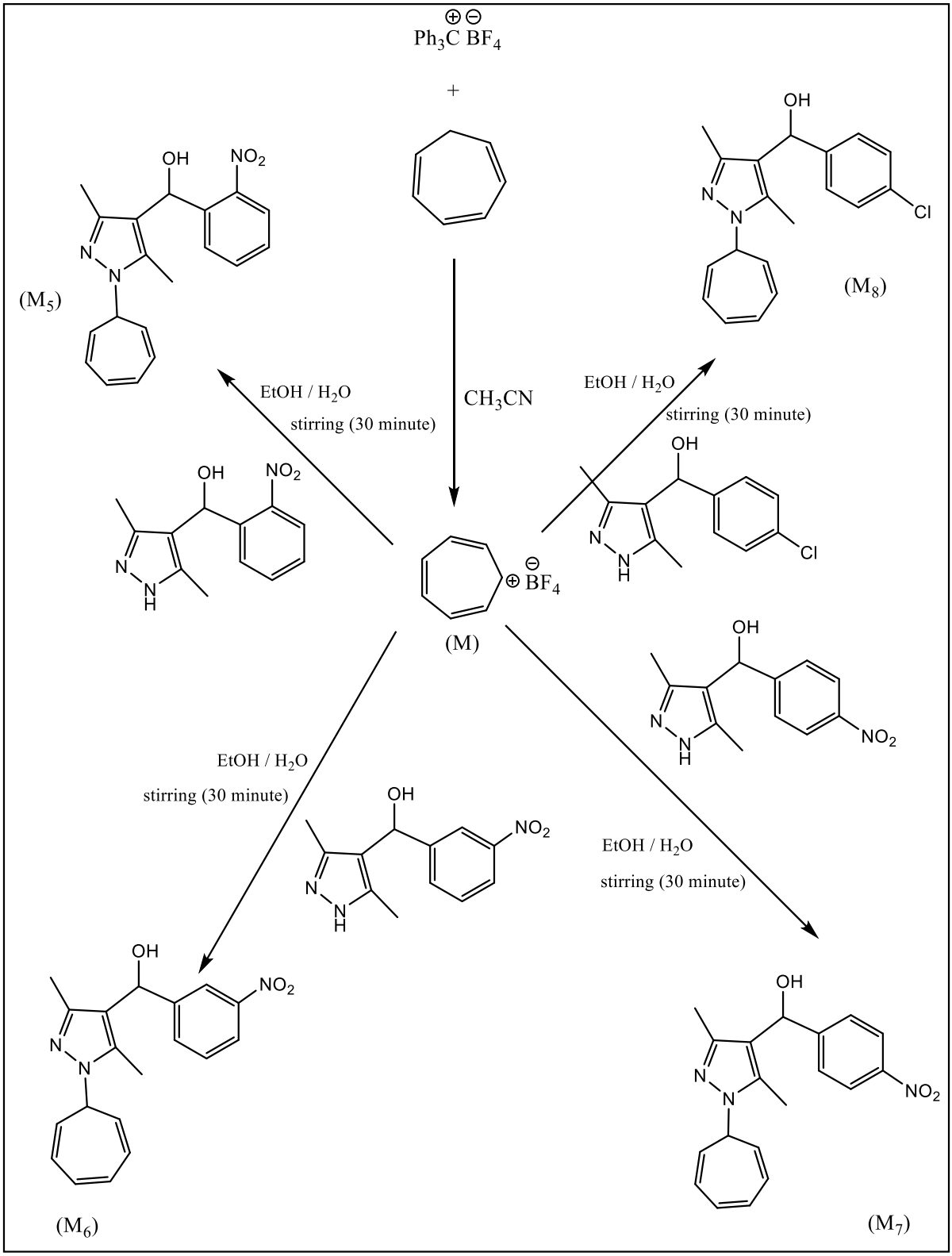
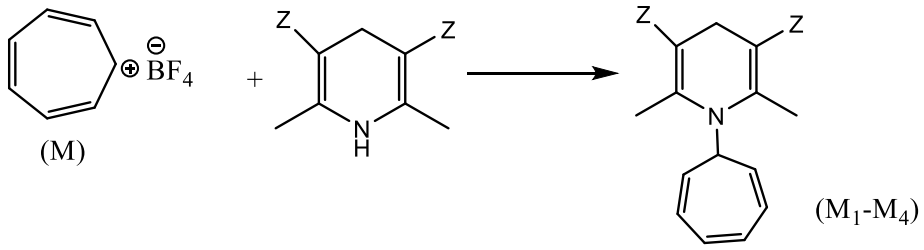


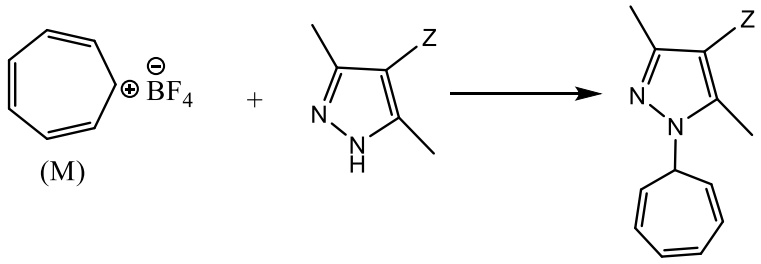
SCHEME 2: synthesis of tropylatedpyrazole derivatives (M_5-M_8)

RESULTS AND DISCUSSION

Previously [29], we achieved preparation of tropyliumtetrafluroborate (M). In this work, we achieved N-tropylation of some dihydropyridines and pyrazoles via simple protocol, easy of workup and mild conditions:



Z= acetyl
(3-yl)ethylidene)hydrazine-1-carboxamide
3-(1-(2-phenylhydrazineylidene)ethyl)
3-(1-hydrazineylideneethyl)



Z= ((2-nitrophenyl)-4-yl)methanol
((3-nitrophenyl)-4-yl)methanol
((4-nitrophenyl)-4-yl)methanol
((4-chlorophenyl)-4-yl)methanol

FT-IR spectra

FT-IR stretching bands for compound (M_2) revealed the appearance of (NH_2) at 3384, 3363 cm^{-1}, aromatic (-C-H) at 3028cm^{-1},aliphatic (-C-H) at 2912cm^{-1}, (-C=O) at 1633cm^{-1}, (-C=N-) at 1591cm^{-1} and (-C=C-) at 1502 cm^{-1} as listed in Table-3 and in Chart-1. FT-IR spectrum for compound (M_4) reveled the appearance of (NH_2) at 3386, 3363 cm^{-1}, aromatic (-C-H) at 3141cm^{-1}, aliphatic (-C-H) at 2950cm^{-1}, (-C=N-) at 1635cm^{-1} and (-C=C-) at 1591 cm^{-1} as listed in Table-3 and in Chart-2. FT-IR spectrum for compound (M_5) reveled the appearance of a broad (-OH) at 3394 cm^{-1}, aromatic (-C-H) at 3005cm^{-1}, aliphatic (-C-H) at 2920cm^{-1}, (-C=N-) at 1693cm^{-1}, aromatic (-C=C-) at 1633, 1585$cm^{-1}$and (-NO_2) at 1520,1360 as listed in Table-3 and in Chart-3.FT-IR spectrum for compound (M_6) reveled the appearance of a broad (-OH) at 3678 cm^{-1}, aromatic (-C-H) at 3022 cm^{-1}, aliphatic (-C-H) at 2960 cm^{-1}, (-C=N-) at 1610 cm^{-1} , aromatic (-C=C-) at 1589, 1512 cm^{-1} and (-NO_2) at 1560,1363 as listed in Table-3 and in Chart-4. [30]

Micro elemental analysis for $C_{18}H_{21}NO_2$ (M_1): %C found (76.19) calc. (76.30), %H found (7.44) calc. (7.74), %N found (4.50) calc. (4.94).Micro elemental analysis for $C_{18}H_{25}N_5$ (M_4): %C found (69.25) calc. (69.42),%H found (8.01) calc. (8.09), %N found (22.37) calc. (22.49).Micro elemental analysis for $C_{19}H_{19}N_3O_3$ (M_7):%C found (67.54) calc. (67.64),%H found (5.60) calc. (5.68), %N found (12.39) calc. (12.46).

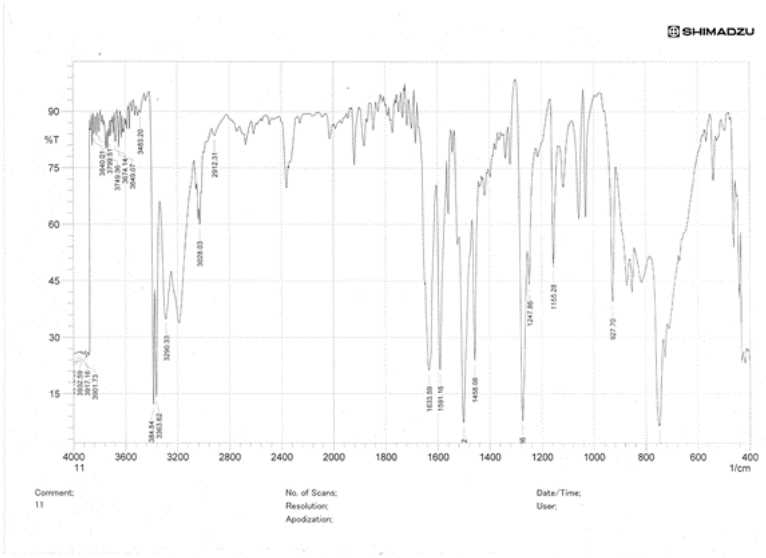


CHART-1 FT-IR for compound M_2

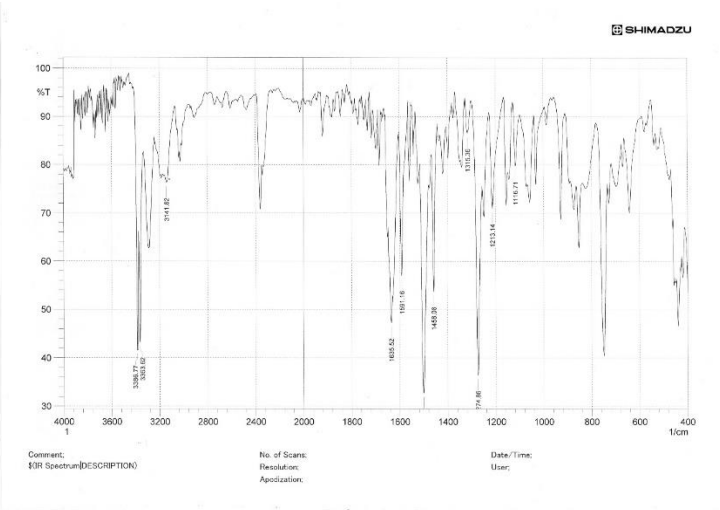


CHART-2 FT-IR for compound M_4

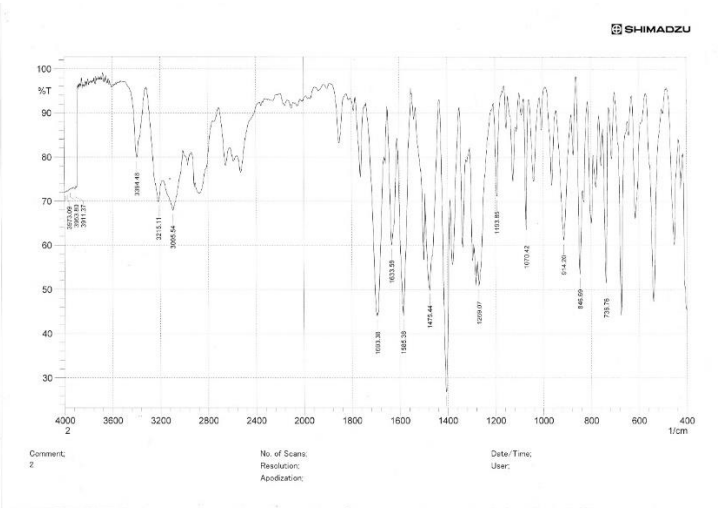


CHART-3 FT-IR for compound M_5

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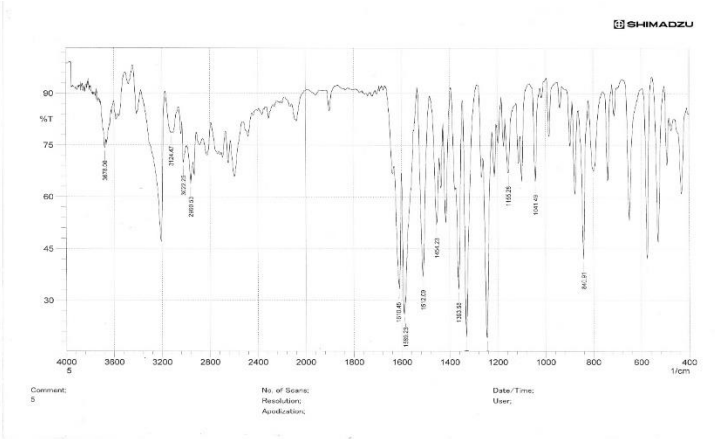

CHART-4 FT-IR for compound M_6

^{13}C-NMR spectra

The ^{13}C-NMR for compound (M_1) in (DMSO as a solvent) revealed the following characteristic signals: C=O (j) at δ170.17 ppm, carbons pointed as (i) at δ166.09 ppm, carbons pointed as (h) at δ 157.25 ppm, carbons pointed as (g) at δ 131.59 ppm, carbons pointed as (f) at δ 127.49 ppm, carbons pointed as (e) at δ 122.57 ppm.

The most important carbon signal pointed as (d) at δ 60.78 ppm, carbon pointed as (c) at δ 35 ppm, carbons pointed as (b) at δ 19 ppm, carbons pointed as (a) at δ 14.70 ppm. (Chart-5) [30].

The ^{13}C-NMR for compound (M_4) revealed the following characteristic signals: carbons (j) at δ 172.98 ppm, carbons pointed as (i) at δ 169.73 ppm, carbons pointed as (h) at δ156.85 ppm, carbons pointed as (g) at δ131.14 ppm, carbons pointed as (f) at δ127.02 ppm, carbons pointed as (e) at δ 122.45 ppm. The most important carbon signal pointed as (d) at δ 60.33 ppm, carbon pointed as (c) at δ 35 ppm, carbons pointed as (b) at δ 22.46 ppm, carbons pointed as (a) at δ 14.25 ppm. (Chart-6) [30].

The ^{13}C-NMR for compound (M_7) revealed the following characteristic signals:carbons (n) at δ170.55 ppm,carbons (m) at δ169.78 ppm,carbon (L) at δ157.88 ppm, carbon (k) at δ153.19ppm.

Carbons pointed as (j) at δ152.82 ppm, carbons pointed as (i) at δ128.75 ppm, carbons pointed as (h) at δ124.20 ppm, carbons pointed as (g) at δ117.96 ppm, carbons pointed as (f) at δ112.56 ppm, carbon pointed as (e) at δ105.38 ppm. The most important carbon signal pointed as (d) at δ61.95 ppm, carbon pointed as (c) at δ33 ppm, carbon pointed as (b) at δ22.69 ppm, carbonpointed as (a) at δ 11.98 ppm. (Chart-7) [30].

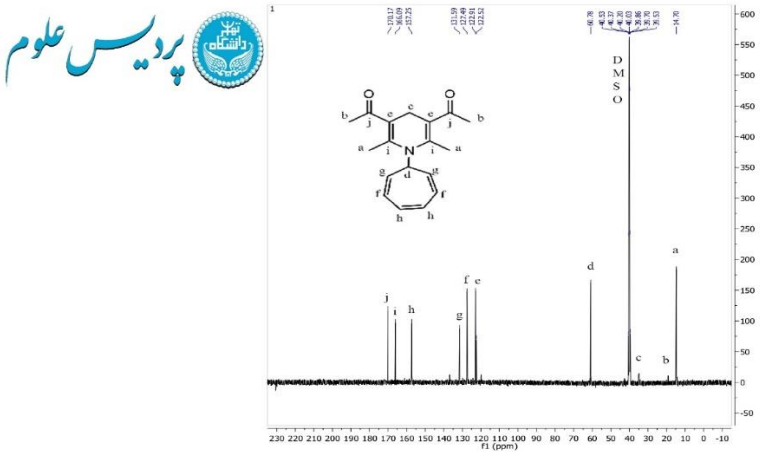

CHART-5^{13}C-NMR for compound M_1

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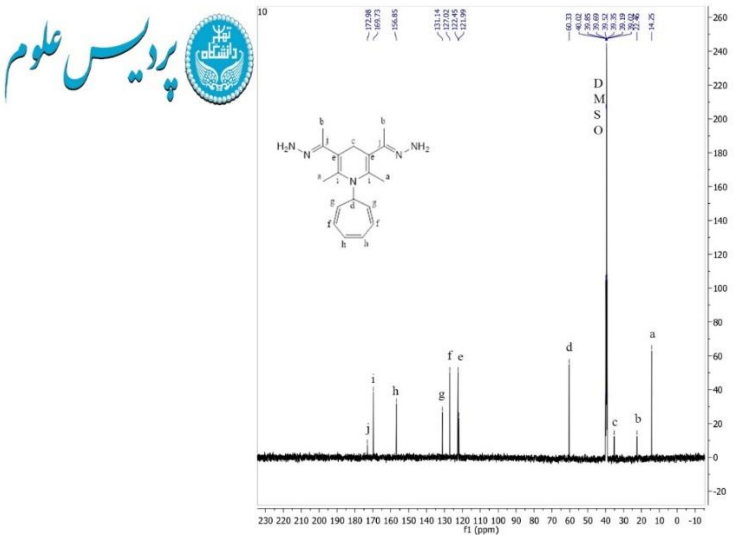

CHART-6^{13}C-NMR for compound M_4

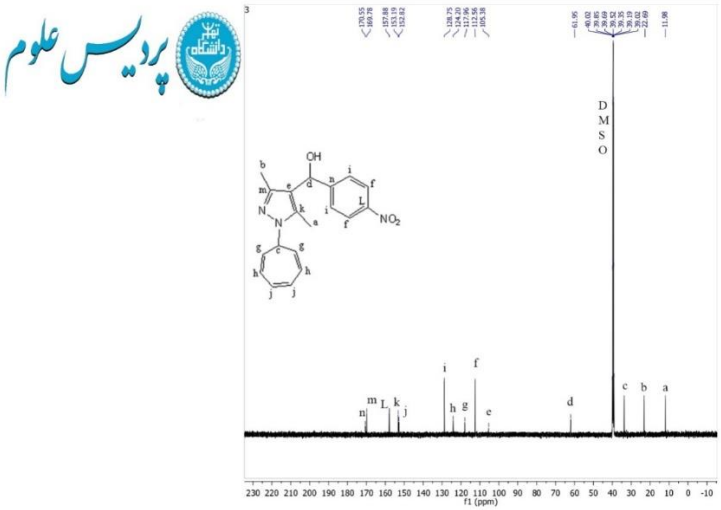

CHART-7^{13}C-NMR for compound M_7

CONCLUSION

In this contribution, we achieved a facile, efficient protocol and good yield to gain newly tropylated five and six heterocyclic moieties. The synthesized compounds identified via (TLC, melting points, FT-IR, ^{13}C-NMR, CHNS elemental analysis).

The selected derivatives (M_2, M_3, M_5, M_7) were testified for (Gram Positive) bacteria, Staphylococcus aureus, Bacillus subtilis, Bacillus pumilusantibacterial activity by Wells method. The results revealed powerful activity against Staphylococcus aureus, and comparable activity against Bacillus subtilis and Bacillus pumilus by comparison with Neomycin as standard drug at the employed concentrations.

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