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Microwave Synthesis of some new compounds 1,3 – Oxazepine – 4 , 7 - dione,derivatives from Schiff bases

Abed M. Daher

Tikrit University -Pharmacy College.

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ABSTRACT

The Microwave technique is a new method for synthesis of organic compounds, which take a pure and high percent of products and completed with short time, compared with conventional method (heating method). The prepared Schiff bases allowed to react by Microwave technique which is a new apparatus for synthesis the organic compounds, reacts with Malic anhydride to yield (1, 3, 6, 7) 1,3 – Oxazepine compounds and reacted with phthalic anhydrides to yield (2, 4, 5, 8) 1,3 – Oxazepine compounds. The useful thing in Microwave technique is using without solvent there for it called (dry method). The synthesized compounds were identified using Melting point apparatus, Infrared Spectroscopy.

Introduction:

Microwave technique method is appears lastly which employed wildly as a method instead of the (Refluxing method) in organic synthesis, because this method is effective and economic, there for it called (MAOS) method, by this method we have high percent and high purity of product compounds with chosen reactions, and the reacts completed with very short time compared with conventional methods.

Microwave method acts by mechanical (Ionic motion) which generated by the passed electrical field in chemical reaction. When we increase the power of Microwave radiation the heat of the perimeter of reaction increase too, these for the conductance energies more effective especially when the reaction is Ionic material this cause absorption for the Microwave rapidly, and cause chemical reaction soon [1]

Oxazepine compounds are seven – membered heterocyclic unsaturated ring. This ring contain five carbon atoms and one oxygen atom, and one nitrogen atom too, there are three isomers for Oxazepine compounds 1, 2 and 1, 3 and 1, 4 – Oxazepine [2].

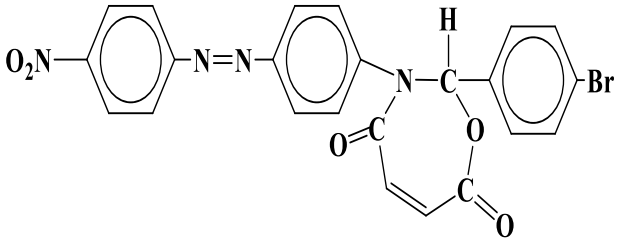
There are different ways for prepare Oxazepine seven – membered heterocyclic ring, for example direct addition of malic anhydride and phthalic anhydride and succinic anhydride to double bond ($-\overset{|}{C}=N-$) for Schiff bases [3].

The Microwave is an electromagnetic radiation with frequency about (0.3 to 300 GHz) [4 – 5].

The principle of Microwave in chemical reactions it is by heating the chemical material with effect of (thermal Microwave constant) this phenomena depend on specific ability for material to absorption of Microwave energy, which by this (heating) can be converts to a new chemical material and new bonds between atoms [6].

Synthesized Compounds (1 – 8)

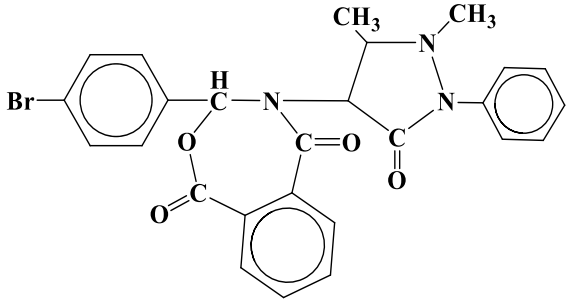
1-



2-(4-bromophenyl)-3-(4-((4-nitrophenyl)diazenyl)phenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione

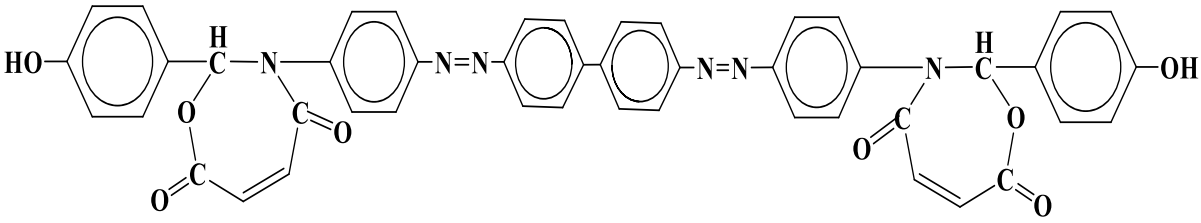
* Corresponding author at: Tikrit University -Pharmacy College.E-mail address:

2-



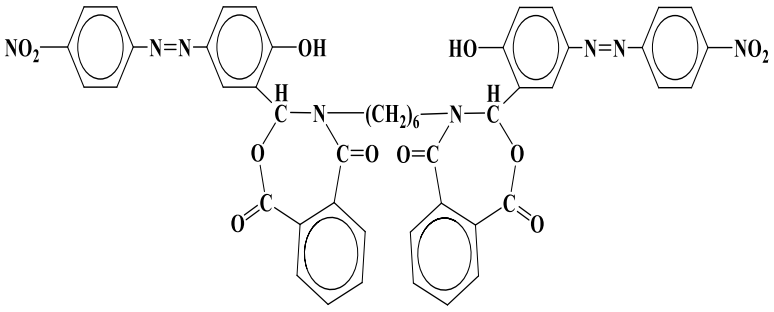
3-(4-bromophenyl)-4-(1,5-dimethyl-3-oxo-2-phenylpyrazolidin-4-yl)
-3,4-dihydrobenzo[*e*][1,3]oxazepine-4,7-dione

3-



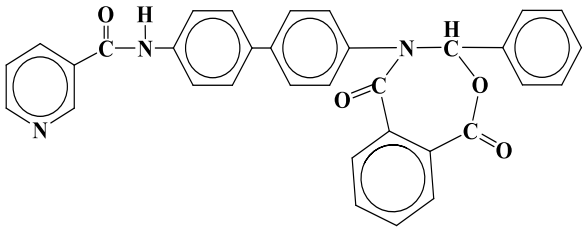
3,3'-(4,4'-(biphenyl-4,4'-diylbis(diazene-2,1-diyl))bis(4,1-phenylene))
bis(2-(4-hydroxyphenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione)

4-



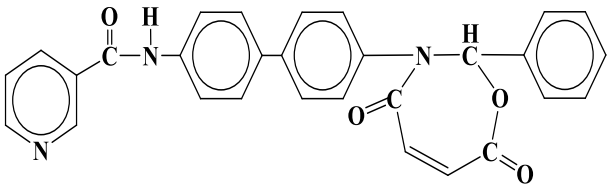
4,4'-(hexane-1,6-diyl)bis(3-(2-hydroxy-5-((4-nitrophenyl)diazenyl)
phenyl)-3,4-dihydrobenzo[*e*][1,3]oxazepine-4,7-dione)

5-



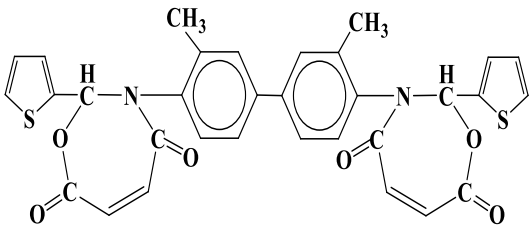
N-(4'-(-3-phenylbenzo[*e*][1,3]oxazepin-4(1*H*,3*H*,5*H*)-yl)biphenyl-4-yl)nicotinamide

6-



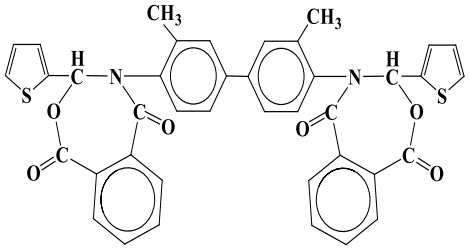
N-(4'-(4,7-dioxo-2-phenyl-1,3-oxazepin-3(2*H*,4*H*,7*H*)-yl)biphenyl-4-yl)nicotinamide

7-



3,3'-(3,3'-dimethylbiphenyl-4,4'-diyl)bis(2-(thiophen-2-yl)-2,3-dihydro-1,3-oxazepine-4,7-dione)

8-



4,4'-(3,3'-dimethylbiphenyl-4,4'-diyl)bis(3-(thiophen-2-yl)-3,4-dihydrobenzo[*e*][1,3]oxazepine-4,7-dione)

Experimental:

Materials:

All materials were from Aldrich and were used further purification.

Instruments:

a. Microwellengrate 8020 (privilege).
b. FT. IR Spectrophotometer Model Shimadzu 8400.
c. Melting point apparatus Model Gallen Kamp (11H).

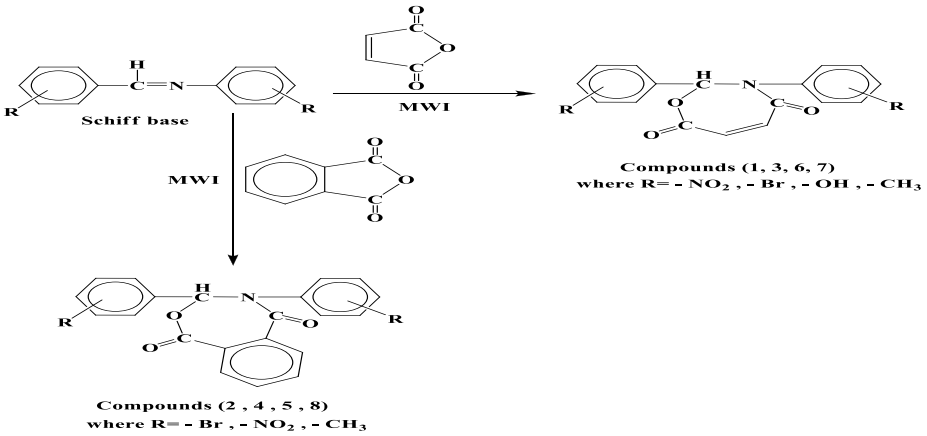
Synthesis of the 1,3 – Oxazepine compounds

a. Take (0.01 mol) from prepared sutabial Schiff base, mixed and crashed with (0.01 mol) of dry Malic anhydride and irradiated by Microwave technique for (6, 5, 6, 5) minutes yield the compounds (1, 3, 6, 7) respectively, the products cool and recrystallized by ethanol.
b. Take (0.01 mol) from prepared sutabial Schiff base, mixed and crashed with (0.01 mol) of dry phthalic anhydride and and irradiated by Microwave technique for (20, 3, 10, 5) minutes yield the compounds (2, 4, 5, 8) respectively, the products cool and recrystallized by ethanol.

Physical properties for the prepared compounds

Comp No.	Schiff bases parent starting material M.P C°	Colour	Moleculer Formula	Yield %	M.P C°	M. WI Power (Waat)	React time / minute	Recrystlization Solvent
1	138 – 140	Yellow powder	$C_{23}H_{15}N_4O_2Br$	85	160 – 162	80 w	6	Abs Ethanol
2	254 – 259	Yellow powder	$C_{25}H_{21}N_3O_4Br$	90	196 – 198	360 w	20	Abs Ethanol
3	140 – 144	Black powder	$C_{14}H_{32}N_6O_8$	86	175 – 180	510 w	5	Abs Ethanol
4	128 – 130	Brown powder	$C_{48}H_{38}N_8O_{12}$	75	255 – 259	520 w	3	Abs Ethanol
5	220 – 223	Yellow powder	$C_{33}H_{23}N_3O_4$	89	284 – 286	360 w	10	Abs Ethanol
6	220 – 222	White powder	$C_{29}H_{21}N_3O_4$	95	225 – 228	360 w	6	Abs Ethanol
7	130 – 135	Orange powder	$C_{32}H_{24}N_2O_6S_2$	80	181 – 184	180 w	5	Abs Ethanol
8	130 – 135	Yellow powder	$C_{40}H_{28}N_2O_6S_2$	50	164 - 168	510 w	5	Abs Ethanol

The reaction scheme:



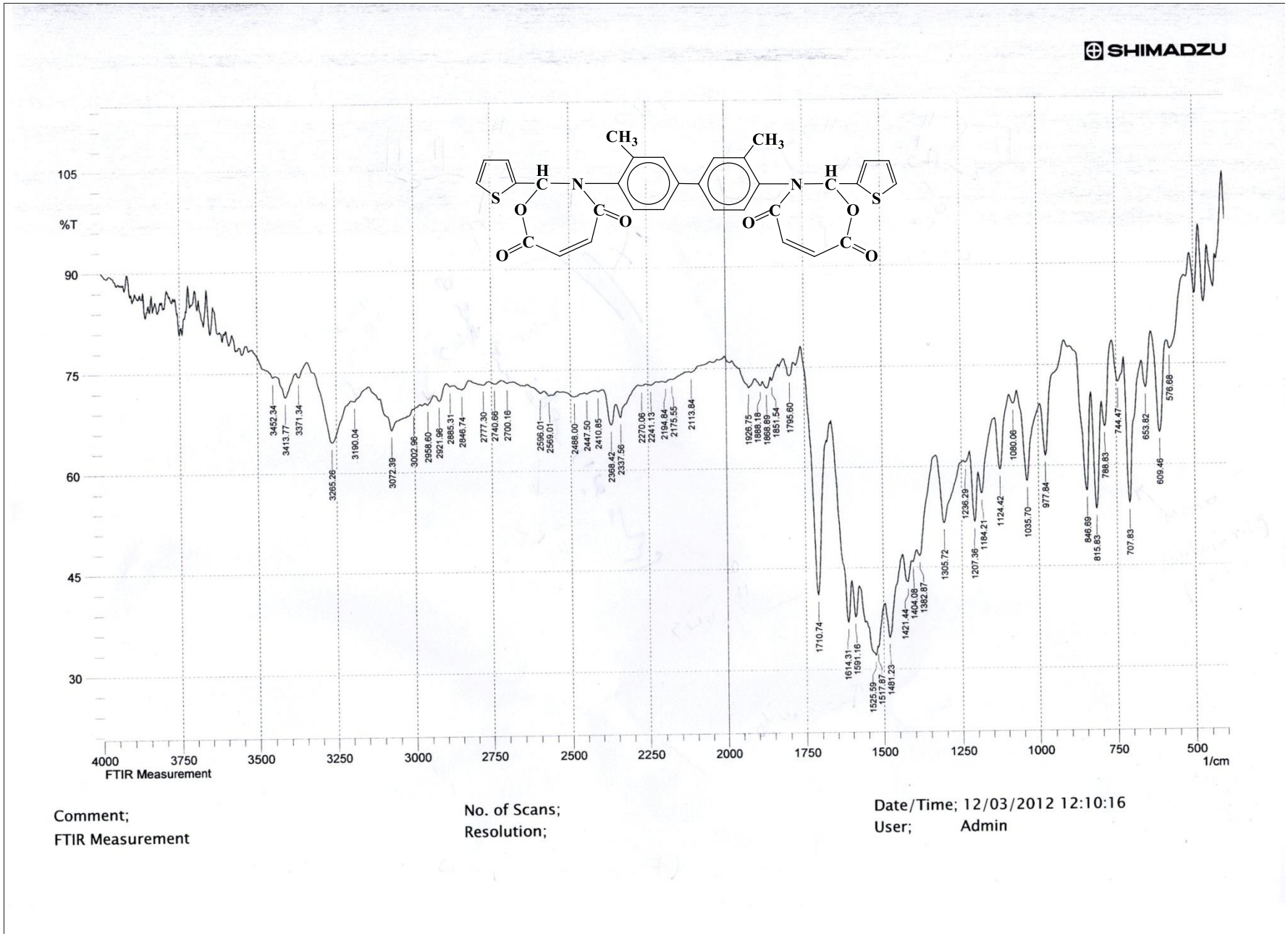
Result and discussion

The synthesis compounds by (Microwave technique) started from the aromatic Schiff bases to produce the (1 – 8) 1,3 – Oxazepine -4, 7 dione compounds and 1,3 – Oxazepine -, 5- dione. FT. IR. Spectral data showed the bands of the functional groups that substituted in rings or in others.

Because the more resembles between the compounds, we take the compounds (4) & (7) as a sample. The bands at 1585 cm^{-1} for azo. groups ($-N=N-$) in (1, 3, 4) compounds.

The observed bands at (1338 & 1404) cm^{-1} for ($-NO_2$) groups. The lactam ($-\overset{O}{\overset{\|}{C}}-N$) bands observed at 1691 cm^{-1}, and the phenol ($-OH$) group observed at 3456 cm^{-1}, the thioether ($-S-$) bands observed at 259 cm^{-1}, the aromatic ($-C=C-$) bands observed at (159 –1600) cm^{-1}, the mono ($-CH_3$) substitution on the phenyl rings in compound (7) bands observed at 707cm^{-1}.

Finally the bands of (**C–H**) aryl and (**C–H**) aliphatic observed at 3190 cm^{-1} and 2958 cm^{-1} respectively [7].


SHIMADZU
%T
105
90
75
60
45
30
4000
3750
3500
3250
3000
2750
2500
2250
2000
1750
1500
1250
1000
750
500
1/cm
FTIR Measurement
3452.34
3413.77
3371.34
3265.26
3190.04
3072.39
3002.96
2958.60
2921.96
2885.31
2846.74
2777.30
2740.66
2700.16
2596.01
2569.01
2488.00
2447.50
2410.85
2368.42
2337.56
2270.06
2241.13
2194.84
2175.55
2113.84
1926.75
1888.18
1868.89
1851.54
1795.60
1710.74
1614.31
1591.16
1525.59
1517.87
1481.23
1421.44
1404.08
1382.87
1305.72
1236.29
1207.36
1184.21
1124.42
1080.06
1035.70
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815.83
788.83
744.47
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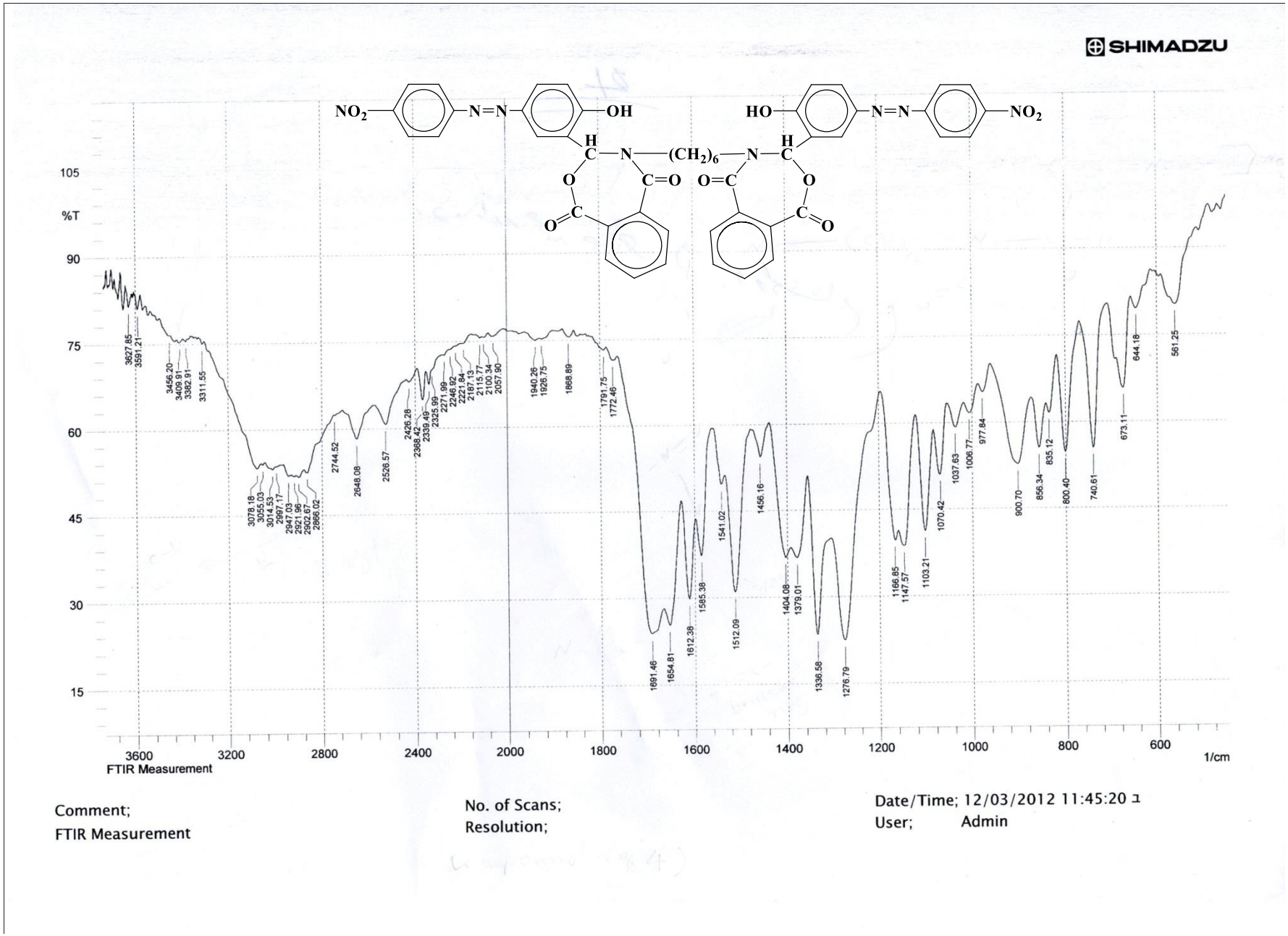
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OH
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NO2
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(CH2)6
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C=O
O=C
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105
%T
90
75
60
45
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15
3627.85
3591.21
3456.20
3409.91
3382.91
3311.55
3078.18
3055.03
3014.53
2997.17
2947.03
2921.96
2903.67
2866.02
2744.52
2648.08
2526.57
2426.28
2368.42
2339.49
2325.99
2271.99
2246.92
2221.84
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2111.77
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2057.90
1940.26
1926.75
1868.89
1791.75
1772.46
1691.46
1654.81
1612.38
1585.38
1541.02
1512.09
1456.16
1404.08
1379.01
1336.58
1276.79
1166.85
1147.57
1103.21
1070.42
1037.63
1006.77
977.84
900.70
856.34
835.12
800.40
740.61
673.11
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تحضير بعض المشتقات الجديدة لمركبات 1 ، 3 – أوكسازبين – 4 ، 7 – دايون من قواعد شيف المحضرة مسبقاً بواسطة تقنية المايكروويف

عبد محمد ظاهر

الخلاصة

أن تقنية المايكروويف هي تقنية جديدة على الساحة البحثية العلمية وخاصة في تحضير المركبات العضوية ، ويمكن بواسطة هذه التقنية الحصول على نسبة عالية ونقية من المنتوج بالإضافة الى وقت قصير جداً مقارنةً بالطريقة القديمة التي كانت ستخدم في التحضير العضوي تم مفاعلة قواعد شيف المحضرة مسبقاً ، والتي استخدمت في تحضير المركبات العضوية حديثاً إذ تم مفاعلتها مع انهدريد الماليك لإنتاج مشتقات (1 ، 3 ، 6 ، 7) 1 ، 3 – أوكسازبين – 4 ، 7 – دايون الجديدة ، كما تم مفاعلتها مع انهدريد الفثاليك لإنتاج مشتقات (2 ، 4 ، 5 ، 8) 1 ، 3 – أوكسازبين – 4 ، 7 – دايون الجديدة ايضاً . والمفيد في تقنية المايكروويف أنها تتم بدون استخدام المذيب ولذا فهي تسمى بالطريقة الجافة . المركبات المحضرة تم تشخيصها بواسطة طيف (I.R) الأشعة تحت الحمراء ، ودرجة الانصهار .