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Synthesis Aspects and Data Analysis of Bisthiadiazolines

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Abstract: Present work contains Synthesis Aspects and Data Analysis of bisthiadiazolines. The main purpose of the study is to study the effect of internal spanner length upon the formation of bisthiadiazolines and to find the utilities of bisthiosemicarbazones in the synthesis of bisheterocyclic compounds.

Keywards: hetero atam, bisthiazolines, bisheterocyclic compounds

1. Introduction: Heterocyclic compounds have been studied with major interest in the past decades. Five membered atomic system having three hetero atoms at the symmetric positions have been studied because of their properties that directly influence society. This type of heterocyclic derivatives has large number of pharmaceutical and industrial importance. Thiadiazolines, oxadiazolines possesses a wide range of biological properties such that antitumor, anticancer, antiviral, antibacterial and antihypertensive. For further related study one may refer [1-14].

2. Practical work/Experiment:

1. Synthesis of 4, 4-(ethane-1, 2-diylbis(oxy) dibenzaldehyde 4.20

A mixture of 4 hydroxybenzaldehyde (1.0g, .01 mol) and KoH (.40g, .01 mol) is dissolved in alcohol (40 ml) 1,2- dibromoethane (.70 g, .003 mol) was added. The resulting mixture was refluxed for 3 hours. The solvent was removed and poured into iced HCL. The product was allowed to cool and crystallized from MeoH to give pure compound 4.20.

2. Synthesis of 4, 4-(propane-1, 3-diylbis (oxy)) dibenzaldehyde 4.21

It is prepared by reacting 4-hydroxybenzaldehyde (1.00g, .01 mol) with 1, 3-dibromopropane (.75g, .003 mol). The mixture is refluxed for 2 hours and allowed to cool at room temperature. It is crystallized to give the compound 4.21.

3. Synthesis of 4, 4-(butane 1, 4-diylbis(oxy)) dibenzaldehyde 4.22

It is obtained by reacting 4-hydroxybenzaldehyde (.80g, .01 mol) with 1, 4-dibromobutane. The mixture is crystallized to obtain the compound 4.22.

4. Synthesis of 4, 4-(pentane 1, 5-diylbis(oxy)) dibenzaldehyde 4.23

Compound is synthesized by the reactions of 4-hydroxybenzaldehyde (1.0 g, .01 mol) with 1, 5-dibromopentane (.95g, .003 mol). The mixture is crystallized to obtain the compound 4.23.

5. Synthesis of 4, 4-(hexane-1, 6-diylbis(oxy))dibenzaldehyde 4.24

It is obtained by the reactions of 4-hydroxybenzaldehyde (.85g, .01 mol) with 1, 6-dibromohexane (1.00g, .003 mol) under the similar conditions as 4.20.

6. Synthesis of 4, 4-(octane-1, 8dihylbis(oxy)) dibenzaldehyde 4.25

It is produced by treating 4-hydroxybenzaldehyde (1.00g, .01 mol) with 1, 8-dibromooctane (1.00g, .003 mol) under the similar conditions as 4.20.

7. Synthesis of 1, 2-bis [2-benzylidene-hydrazinecarbothioamide-4-oxy] ethane 4.8

A mixture of compound 4.20 (.80 g, .0032 mol) and thiosemicarbazide .578g, .0056 mol) in dry EtoH (20 ml) and HCL(1.0 ml) is refluxed for 4 hours. After cooling the mixture at room temperature solid is separated. The resulting product was crystallized from MeoH to give 4.8.

8. Synthesis of 1, 3-bis[2-benzylidene-hydrazinecarbothioamide]-4-oxy] propane 4.9

A mixture of 4.21 (.75g, .0028 mol) with thiosemicarbazide (.540g, .0068 mol) is treated under the similar conditions as 4.8.

9. Synthesis of 1, 4-bis[2-benzylidene-hydrazinecarbothioamide]-4-oxy] butane 4.10
It is synthesized by the mixture of 4.22 (.80g, .0029 mol) with thiosemicarbazide (.618g, .0062 mol) under the conditions similar to 4.8.

10. Synthesis of 1, 5-bis[2-benzylidene-hydrazinecarbothioamide]-4-oxy] pentane 4.11
It was crystalized by reacting the mixture of 4.23 (.70g, .0028 mol) with thiosemicarbazide (.482 g, .0061 mol) under the conditions similar to 4.8.

11. Synthesis of 1, 6-bis[2-benzylidene-hydrazinecarbothioamide]-4-oxy] hexane 4.12
It is obtained by mixture of 4.24 (.90g, .0027 mol) and thiosemicarbazide (.482 g, .0057 mol) under the conditions similar to 4.8.

12. Synthesis of 1, -bis[2-benzylidene-hydrazinecarbothioamide]-4-oxy] propane 4.13.
The compound is obtained by the reactions of 4.25 (.90g, .0026 mol) with thiosemicarbazide (.0417g, .0051 mol) under the conditions similar to 4.8.

13. Synthesis of N,N'-(5,5-(4,4-ethane-1,2-diylbis(oxy)bis (4, 1-penylene)) bis(4-acetyl-4, 5-dihydro-1, 3, 4 -thiadiazole- 5, 2 diyl) diacetamide 4.14
A mixture of compound 4.8 (.75g, .0021 mol) and acetic anhydride (20 ml) was refluxed for 7 hours. The resulting mixture was poured in to ice to obtain the solid mixture. It was crystallized from MeoH to obtain pure 4.14.

14. Synthesis of N,N'-(5,5-(4,4-propane-1,3-diylbis(oxy)bis (4, 1-penylene)) bis(4-acetyl-4, 5-dihydro-1, 3, 4 -thiadiazole- 5, 2 diyl) diacetamide 4.15
It is prepared from the reactions of 4.9 (.80g, .0021 mol) with acetic anhydride (20 ml) under the conditions similar to 4.19.
15. Synthesis of N,N'-(5,5-(4,4-butane-1,4-diylbis(oxy)bis (4, 1-penylene)) bis(4-acety l-4, 5-dihydro-1, 3, 4 -thiadiazole- 5, 2 diyl) diacetamide 4.16
It is prepared from the mixture of 4.10 (.90g, .0024 mol) with acetic anhydride (20 ml) under the conditions similar to 4.14.
16. Synthesis of N,N'-(5,5-(4,4-pentane-1,5-diylbis(oxy)bis (4, 1-penylene)) bis(4-acetyl-4, 5-dihydro-1, 3, 4 -thiadiazole- 5, 2 diyl) diacetamide 4.17
It is obtained from the reactions of 4.11 (.70g, .0025 mol) with acetyl hydride (17 ml) under the conditions similar to 4.14.
17. Synthesis of N,N'-(5,5-(4,4-hexane-1,6-diylbis(oxy)bis (4, 1-penylene)) bis(4-acetyl-4, 5-dihydro-1, 3, 4 -thiadiazole- 5, 2 diyl) diacetamide 4.15
It is obtained from the reactions of 4.12 (.85g, .0019 mol) with acetic hydride (27 ml) under the conditions similar to 4.14.
18. Synthesis of N,N'-(5,5-(4,4-octane-1,8-diylbis(oxy)bis (4, 1-penylene)) bis(4-acetyl-4, 5-dihydro-1, 3, 4 -thiadiazole- 5, 2 diyl) diacetamide 4.15
It is prepared from the reactions of 4.13 (.95 g, .0021 mol) with acetic anhydride (26 ml) under the conditions similar to 4.14.

Physical and characteristics spectral data:

Physical and characteristics spectral data of dibenzaldehydes 4.20-4.25

Compound	M.P. (℃)	Yield %	IR (CM^{-1}) C=O	1_{HNMR} CHO	CNMR C=O
4.20	108-110	60	1650	9.74	180.87
4.21	120-124	64	1640	9.80	180.07
4.22	90-92	70	1631	9.17	180.81
4.23	74-76	63	1670	9.08	180.92
4.24	100-102	71	1670	9.08	180.92
4.25	80-83	62	1668	9.01	180.90

Table 4.2 Physical and characteristics spectral data of bisthiosemicarbazones 4.8-4.13

Compound	M.P. (℃)	Yield %	IR (CM^{-1})		1_{HNMR}		CNMR	
			C=N	C=S	NH	NH	C=S	C=N
4.8	128-130	61	1591	1141	7.21	7.17	173.87	146.71
4.9	176-180	80	1589	1171	7.04	7.27	173.82	146.85
4.10	170-173	78	1591	1168	7.24	7.31	173.91	146.97
4.11	168-170	79	1601	1164	7.56	7.32	174.07	146.08
4.12	164-166	71	1590	1140	7.40	7.21	172.08	147.74
4.13	160-162	68	1594	1161	7.34	7.26	171.48	147.94

Table 4.3 Physical and characteristics spectral data of bisthiadiazolines 4.14-4.19

Compound	M.P. (℃)	Yield %	IR (CM^{-1})		1_{HNMR}		CNMR	
			C=O	C=N	1-NH	H-2	1-C=S	2-C=O
4.14	128-130	61	1590	1574	10.87	6.21	167.74	166.98
4.15	130-136	68	1593	1576	10.86	6.34	167.82	166.87
4.16	136-140	70	1593	1581	10.74	6.48	167.94	166.99
4.17	120-123	64	1640	1590	10.90	6.70	168.90	167.40
4.18	140-144	62	1630	1600	10.94	6.71	168.97	167.13
4.19	150-152	69	1636	1604	11.21	6.71	163.81	162.20

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