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# AN ANALYTICAL STUDY ON THE 4-THIAZOLIDINO α, βUNSATURATED KETONES AND DIMETHYL AMINO METHYLENE KETONES

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***Abstract:*** ***In this study we have analyzed about the 4-thiazolidino unsaturated ketones and dimethyl amino methylene ketones. In silica gel-G panels TLC has evaluated the immaculateness of compounds. On Shimadzu FTIR-8400S, IR spectra have been obtained. Dissolvable and synthetic motions are transmitted in ?pm on a sample of 1H NMR at 300 MHz (Bruker) using DMSO-d6. PerkinElmer 4200 Elemental Analyzer was taken using CHNS information. Examples were dried at reduced tension before the study all.***

**Key words-**Thiazolidine, Unsaturated ketones, methylene ketones.

Heterocyclic compounds are the cyclic compounds that contain two or more distinct types of atoms integrated into the ring. There are virtually no limits to the number of potential heterocyclic systems. There are a large number of heterocyclic compounds, and this number is growing fast. There are similarly many literatures in this topic and the latter studies are considerably greater among the three main categories of organic, carbocyclic and heterocyclic chemistry. In Chemical Abstracts, over six million compounds have been documented and almost half are heterocyclic.

The heterocyclic molecules, which play a major part in the metabolism of all living cells, are extremely widespread and necessary for existence. A variety of heterocyclic compounds are pharmacologically active, several are regularly used in clinics. Some of them are natural compounds like penicillin and cephalosporin, alkaloids like vinblastine, ellipticine, morphine and reserpine and cardiac glycosides such as digitalis. These are also natural products. However, the vast majority of synthetic heterocyclics, such as anti-carcer, analgetic, analeptics, hypnotic and vasopressin agent and pesticides, insecticides, weedkillers, and rodenticides, have been widely used.

**LITERATURE REVIEW-** X.Hee tal. [2013] An epic diamine-based benzoxazine monomer containing ester and cumbersome fluorene gatherings (BABPF-p) was effectively incorporated by scientist. The synthesis was done by the reaction of 9,9-bis- [4-(4-amino benzoyloxy) phenyl] fluorene with paraformaldehyde and phenol. The compound construction of monomer was affirmed by IR and 1H and 13C NMR spectroscopic information.

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Dilesh et al. [2013] portrayed that dihydro subsidiaries are steadier than the 1,3-benzoxazines themselves towards hydrolyzing specialists. The compounds of methoxy 1,3-benzoxazine which show the new methodology empowers the synthesis of 2,3-disubstituted-1,3-benzoxazines as the lactone.

A. Van et al. [2014] completed synthesis of benzoxazine pitch from sustainable regular asset vanillin. Scientist blended a benzoxazine and quinazoline ring frameworks in great yield. They received a way of cyclo-condensation of 2?aminobenzamide and salicylamide with aldehydes and ketones utilizing hetero polyacid impetus. The benefits of strategy are perfect reactions, basic workup method and climate cordial conditions.

**OBJECTIVES OF THE STUDY**

* To understand the Structure of compounds.
* To analyze the Spectral data of compounds.
* To analyze the aryl substituted 4-thiazolidino ?, ? unsaturated ketones and dimethyl amino methylene ketones.

**MATERIAL AND METHODS-**

1. Preparation of 4-phenylthiazole-2-amine IK-2014-002a

A solution of IK-2014-001a was added to an ethanol thiourea solution (0.02mol) rapidly at room temperature (0.02mol). The precipitation appeared immediately after the expansion was completed. The suspension was placed over crushed ice and mixed with the NH4OH solution, then washed at room temperature for 30 minutes, and dried water. Logically pure IK-2014-002a was given by ethanol recrystallization. Renders (70.3%) and m.p.145-148°C. In the planning of IK-2014-002b, the same methodology has been maintained.

2. Preparation of 2-chloro-N-(4-phenylthiazol-2-yl) acetamide IK-2014-003a

Chloroethylene chloride (0.02 mol) in 20 ml dry benzene hold at 0,0-50C was gradually supplied with a solution of IK-2014-002a (0.01 mol) and a three-hour blend of reactions reflux. After the

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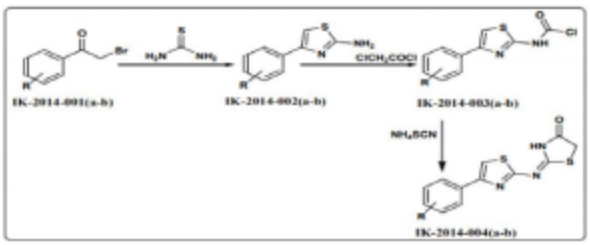
product is constructed in, the raw product has been recrystallized using the solution of NaHCO3 and cold water to create shade of precious compound IC-2014-003a stones. Renders (66.4%) and m.p.184-186oC, in compound readiness IK-2014-003b, the same system was continued.

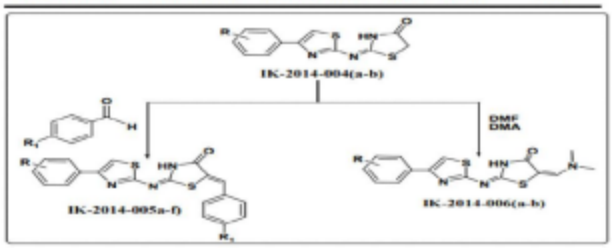
**RESULT AND DISCUSSION-** A study has shown synthesis using bident nucleophiles, such as hydrazine, urea, thiourea and g, active syntheses like ?, ?-unsaturated ketones and Di methylene ketones in the form of syntheses with five, six and seven heterocyclic rings to make it simple to synthesize (to synthesize seven-membered rings). Appropriate reagents in the COCH2 group chemicals in the molecule may readily create the abovementioned compounds.

For [IK-2014-004 (a-b)] and [IK-2014-006(a-b)] the principal composition is the thiazolidin-4-one (E)-2-(4-phenylthiazol-2-ylimino) synthesis [IC-2014-004(a-b)]. The [IK-2014-004(a-b), IK-2014-1 compounds were manufactured. The phenacylic bromides converted by a ClCOCH2Cl reaction to [IK-2014-003(a-b)] were then cycled to thiourea in Scheme-IK-2014-1, under the reaction condition of Hantzsch in order to generate replacement 2-aminothiazoles. NH4SCN cycling was performed [IK-2014-003(a-b)]. [IK-2014-004(a-b)].

As shown in Scheme IK-2014-0024, [IK-2014-006(a-b)], [iK-2014-004(a-b)] and dimethylamine methylene ketones were known to have ?-, ?-unsaturated [IK-2014-005(a-h)] reaction [IC-2014-006(a-b)] respectively.




| Codes of Compounds | | | | Substituent |
|---|---|---|---|---|
| | | | | R |
| IK-2014-001a | IK-2014-002a | IK-2014-003a | IK-2014-004a | H |
| IK-2014-001b | IK-2014-002b | IK-2014-003b | IK-2014-004b | OMe |




| Codes of Compounds | Substituent | | Codes of Compounds | Substituent | | Codes of Compounds | Substituent | |
|---|---|---|---|---|---|---|---|---|
| | R | $R_1$ | | R | $R_1$ | | R | $R_1$ |
| IK-2014-005a | H | Cl | IK-2014-005d | H | Cl | IK-2014-006a | H | H |
| IK-2014-005b | H | Br | IK-2014-005e | H | Br | IK-2014-006b | H | OMe |
| IK-2014-005c | H | OMe | IK-2014-005f | H | OMe | | | |

**Structure of compounds**

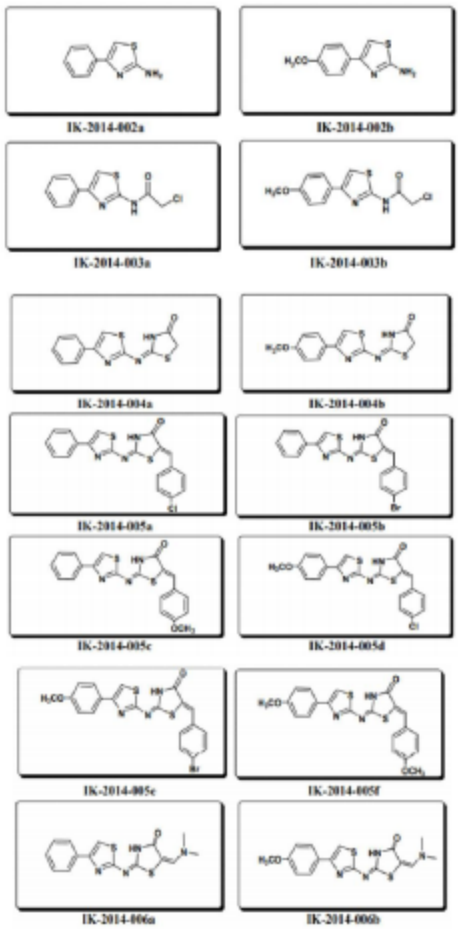



**Table 1: Physical and analytical data of compounds**

| S. No. | Codes of Compounds | Molecular Formula | M.W. | Yield (%) | M.P. (°C) | Elemental Analysis Observed (Theoretical) Percentage | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | C | H | N | S |
| 1. | IK-2014-002a | $C_9H_8N_2S$ | 176.24 | 70.3 | 165-168°C | 60.20 (61.34) | 4.68 (4.58) | 15.45 (15.90) | 17.90 (18.19) |
| 2. | IK-2014-002b | $C_{10}H_{10}N_2OS$ | 206.26 | 72.5 | 195-199°C | 57.77 (58.23) | 4.50 (4.89) | 13.40 (13.58) | 15.01 (15.55) |
| 3. | IK-2014-003a | $C_{11}H_8ClNO_2S_2$ | 253.33 | 66.4 | 184-186°C | 50.84 (51.64) | 3.55 (3.15) | 17.27 (17.86) | 27.04 (27.25) |
| 4. | IK-2014-003b | $C_{12}H_{11}NO_3S_2$ | 265.35 | 62.9 | 186-192°C | 49.50 (49.79) | 4.20 (4.38) | 15.90 (15.64) | 24.09 (24.17) |
| 5. | IK-2014-004a | $C_{12}H_9N_3OS_2$ | 275.35 | 68.0 | 195-198°C | 52.43 (52.34) | 3.30 (3.28) | 15.02 (15.26) | 23.43 (23.29) |
| 6. | IK-2014-004b | $C_{13}H_{11}N_3O_2S_2$ | 305.38 | 70.5 | 205-208°C | 51.00 (51.13) | 3.35 (3.63) | 13.09 (13.76) | 21.23 (21.00) |
| 7. | IK-2014-005a | $C_{18}H_{11}ClN_2OS_2$ | 397.90 | 62.0 | 220-222°C | 57.45 (57.25) | 3.32 (2.84) | 10.45 (10.56) | 16.15 (16.12) |
| 8. | IK-2014-005b | $C_{18}H_{11}BrN_2OS_2$ | 442.35 | 66.0 | 230-232°C | 50.45 (51.59) | 2.66 (2.72) | 5.45 (0.58) | 14.34 (14.53) |
| 9. | IK-2014-005c | $C_{19}H_{14}N_2O_2S_2$ | 393.48 | 71.0 | 265-270°C | 61.32 (61.05) | 3.80 (3.84) | 10.23 (10.68) | 16.21 (16.30) |
| 10. | IK-2014-005d | $C_{18}H_{11}ClN_2O_2S_2$ | 427.93 | 70.0 | 240-245°C | 56.02 (56.13) | 3.27 (3.30) | 5.00 (0.02) | 14.36 (14.90) |
| 11. | IK-2014-005e | $C_{18}H_{11}BrN_2O_2S_2$ | 472.28 | 65.4 | 220-225°C | 50.00 (50.05) | 2.90 (2.98) | 5.76 (0.90) | 13.60 (13.70) |
| 12. | IK-2014-005f | $C_{19}H_{14}N_2O_3S_2$ | 422.51 | 68.0 | 186-188°C | 59.55 (59.36) | 4.11 (4.05) | 5.90 (9.02) | 15.13 (15.16) |
| 13. | IK-2014-006a | $C_{12}H_{10}N_4OS_2$ | 330.43 | 72.4 | 130-135°C | 54.50 (54.22) | 4.22 (4.27) | 16.90 (16.96) | 19.40 (19.43) |
| 14. | IK-2014-006b | $C_{13}H_{12}N_4O_2S_2$ | 360.45 | 68.0 | 125-128°C | 53.20 (53.11) | 4.34 (4.47) | 15.50 (15.54) | 17.75 (17.70) |

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| S. No. | Codes of Compounds | IR (KBr) $cm^{-1}$ | $^1$HNMR ($CDCl_3$ / DMSO-$d_6$) δ ppm | m/z (% abundance) |
|---|---|---|---|---|
| 9. | IK-2014-005c | 3354 (N-H str.), 3037 (C-H str.), 2855 ($OCH_3$), 1656 (C=O), 1585 (C=N str.), 1533 (C=C str.). | 8.13 (1H, s, NH), 8.00 (1H, s, ArH), 7.77 (1H, s, CH), 7.65-7.59 (5H, m, ArH), 7.45-7.20 (4H, m, ArH), 3.73 (3H, s $CH_3$). | 394 (20), 393 (45), 425 (100). |
| 10. | IK-2014-005d | 3354 (N-H str.), 3037 (C-H str.), 2855 ($OCH_3$), 1650 (C=O str.), 1584 (C=N str.), 1533 (C=C str.), | 8.13 (1H, s, ArH), 8.05 (1H, s, NH), 7.80 (1H, s, =CH), 7.55-7.49 (4H, m, ArH), 7.24-7.22 (4H, m, ArH), 3.80 (3H, s, $CH_3$) | 428 (45), 427 (30), 222 (100). |
| 11. | IK-2014-005e | 3354 (N-H str.), 3037 (C-H str.), 2850 ($OCH_3$), 1655 (C=O str.), 1585 (C=N str.), 1533 (C=C str.), | 8.13 (1H, s, ArH), 7.79 (1H, s, NH), 7.60 (1H, s, =CH), 7.55-7.48 (4H, m, ArH), 7.24-7.22 (4H, m, ArH), 3.80 (3H, s, $CH_3$) | 473 (100), 472 (70), 429 (55). |
| 12. | IK-2014-005f | 3334 (N-H str.), 3035 (C-H str.), 2860 ($OCH_3$), 1630 (C=O str.), 1575 (C=N str.), 1533 (C=C str.), 1030 (C-N str.). | 8.00 (1H, s, ArH), 8.05 (1H, s, NH), 7.80 (1H, s, =CH), 7.44 (6H, m, ArH), 7.43 (3H, m, ArH), 3.73 (6H, s, $CH_3$) | 424 (100), 423 (65), 405 (45). |
| 13. | IK-2014-006a | 3354 (N-H str.), 3040 (C-H str.), 2860 ($OCH_3$),1645 (C=O str.), 1580 (C=N str.), 1533 (C=C str.), 1035 (C-N str.). | 8.13 (1H, s, ArH), 8.00 (1H, s, NH), 7.22-7.48 (5H, m, ArH), 6.22( 1H, s, =CH), 3.06 (6H, s, $CH_3$). | 331 (65), 330 (45), 215 (100). |
| 14. | IK-2014-006b | 3340 (N-H str.), 3010 (C-H str.), 2845 ($OCH_3$),1645 (C=O str.), 1589 (C=N str.), 1533 (C=C str.), 1030 (C-N str.). | 8.00 (1H, s, ArH), 7.40 (1H, s, NH), 7.37-6.83 (4H, m, ArH), 6.22( 1H, s, =CH), 3.73 (3H, s, $CH_3$), 2.47 (3H, s, $CH_3$). | 360 (55), 361 (25), 294(100). |

Interpretation of Spectral Data for the Elucidation of Structure of Compounds Structures of all substances have been determined based on the basic examination of IR, 1H NMR and MS spectra data. All physical data of all compounds in the provided structures have been repeatedly verified to be exact. The micro analysis and spectral data are used to provide all substances.

1. InterpretationofspectraldatafortheelucidationofcompoundIK-2014-004a

**IRspectrum:** IK-2014-004a displayed infrared spectrum at 3354 (NH str.); 3037 (C-H str.), 1656 (C=O st); 1584 (C=C str.) cm-1 exhibited at KBr pellets.

**1HNMRspectrum:** 1H In IK-2014-004a, 1 proton is tied to a CH of ? 8.13 ppm, 1 proton of NH is tied to ? 8.0 ppm, 5 of which are tied to one aromatic carbon ring atom in the range ? 7.79 to 7.41 ppm, respectively, were determined to be DMSO-d6 indicator of the existence of 9 protons. At ? 3.84 ppm a singlet was found for 2H. Composite training IK-2014-004a clearly shown,

**MSspectrum:** The ClCH2COOH IK-2014-003a cyclization was tested by the MS spectrum compound IK-2014-004a. The data were discovered in m/z and significant maximum values were 222 (100%), 274 (50%) and 276 (35 %). Likewise, spectral interpretation of the IK-2014-004b compound,

2. InterpretationofspectraldatafortheelucidationofcompoundIK-2014-005c

**IRspectrum:** The infrared spectrum of IC-2014-005c compound shows 3358 (N-H str) and 3037 (C-H str.) peaks, 1656 (C=O str.) and 1584 (C=C str.), cm-1 peaks. Absorption in the 1714 cm-1 and ?,?- unsaturated ketone 1656 cm-1 carbonyl group has been proposed for the production of the compound IK-2014-005c compound

**1HNMRspectra:** 1H In DMSO-d6 signals of 12 proton 1 were seen in the composite spectrum NMR, IK-2014-005c, of which 1 NH was ? 8.0 ppm with 1 singlet to 1 proton 1 with ? 7.80 ppm with ?H bonded with ? 8,13 ppm with 1 NH. In the range ? 7.79-7.25 the molecule IC-2014-005c is clearly visible, and 9 protons are linked to two sweetened rings' carbon atoms.

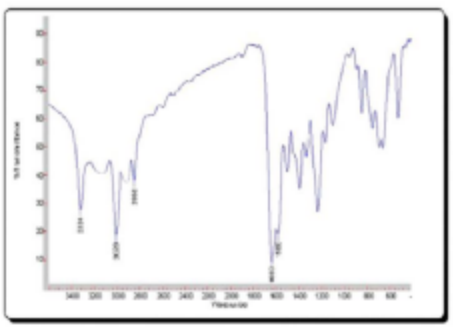
**MSspectrum:** The production of unsaturated ketone in compound IK-2014-004a and benzaldehyde was also verified by MS compound spectrum IK-2014-005c. Values in m/z and large peaks in 370 (100%), 392 (55%) and 394 were found (15%). Simile, the compound IK-2014-005 was determined using spectral interpretations (a-f)

3.Interpretation of spectral data for thee lucidation of compound IK-2014-006a

**IRspectrum:** The IK-2014-006a, infrared spectra was reported to be 3358 (N-H str.), 3037 (C-H str.), 1656 (C=O str.), 1584 (C=C str.), cm-1 at peaks at KBr pellet. The absorption in poor quality dialkyl aryl ketone 1714 cm-1 and intake of 1656 cm-1?, ?- unsaturated ketone indicated the synthesis compounds of IK-2014-006a.

**1HNMRspectra:** 1H The IK-2014-006a NMR compound spectra revealed 14 proton signals, with ? 8.13 ppm, one NH proton at ? 8.0 ppm, five protons with one aromatic ring of carbon atoms detected at ? 7.79-7.46, three singlet 1 singlet for one CH proton observed at ? 6.55 ppm. And a singlet of the two methyl groups was found at ? 3.06 ppm for 6H. Clearly indicated compound IK-2014-006a.

**MSspectrum:** The IK-2014-006a compound MS spectrum has also verified the production of the IK-2014-004a compound dimethyl amino methylene ketone. Walled at 240 (100%), 329 (65%) and 331 are shown in m/z and significant peaks are seen (18%). The shape of compound IK-2014-006b was similarly defined using spectral interpretations.



Spectrum No. 1: (e)-4-(IK-2014-004a) (E)-2-

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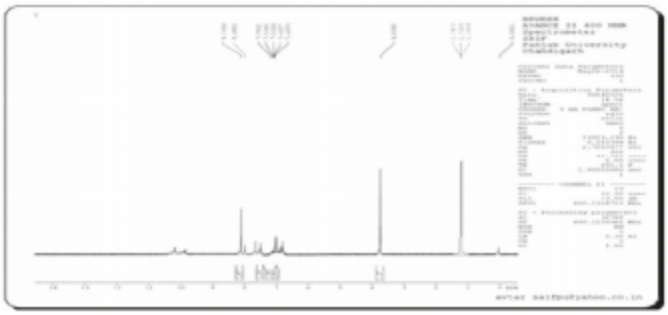
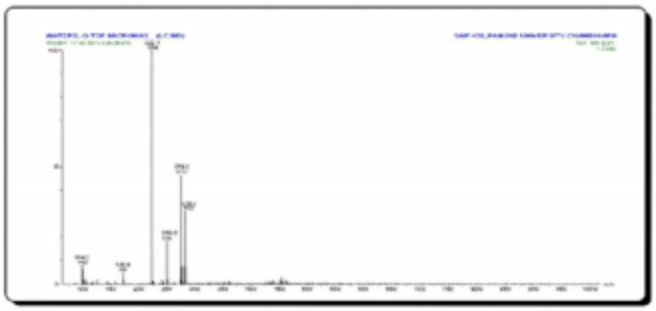
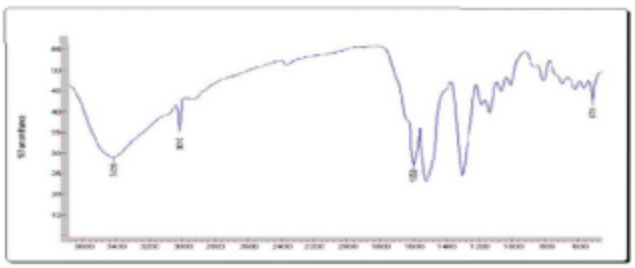
Thiazolidin (4-phenylthiazol-2-ylimino IR spectrum)



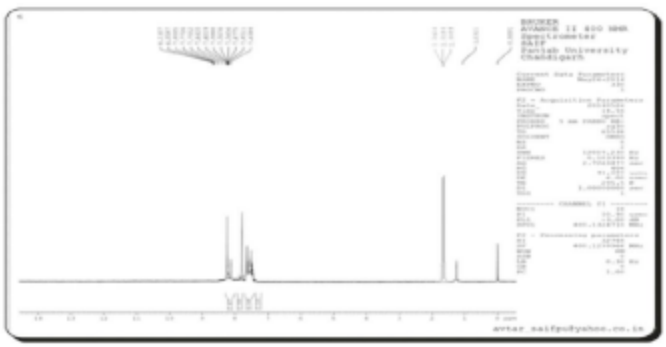
Spectrum No. 2: (1H-NMR thiazolidin-4-one spectrum E)-2-(4-phenylthiazol-2-ylimino) (IK-2014-004a)



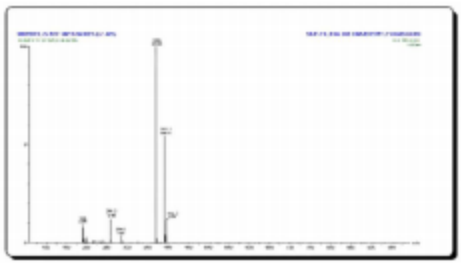
Spectrum No. 3: Thiazolidin-4-one (E)-2-(4-phenylthiazol-2-ylimone) Mass spectrum (IK-2014-004a)



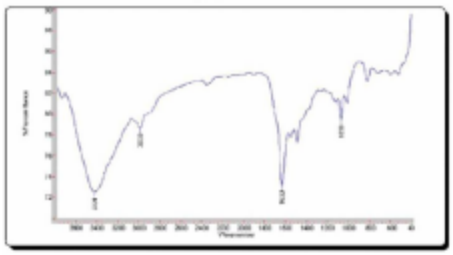
Spectrum No.4: (2E,5Z)-5-(4-methoxybenzylidene)-2-(4-phenylthiasol-2-ylimine) Thiazolidins-4-one IR spectrum (IK-2014-005a)



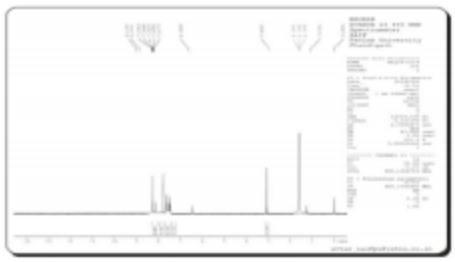
Spectrum No. 5: Thiazolidin-2-(4-phenylthiazol-2-ylimone) HNMR spectrum (2E,5Z)-5-(4-methoxybenzylidene)



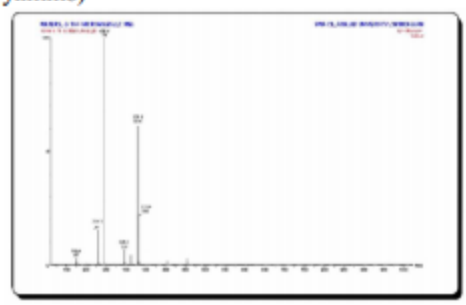
Spectrum No.6: Thiazolid-4-one (2-phenylthiazol-2 ylimino) (2E,5Z)-5-(4-methoxybenzylidene)-2-Spectrum weight (IK-2014-005a)



Spectrum No. 7: IR-spektrum (2e-,5E-5-(dimethylamino) methylene) thiazolidine (4--4-one) (IK-2014-006a IR-spectrum)



Spectrum Number8: 1HNMR (2E,5E)-5-(dimethylamine)-2-(4-phenylthiazol-2-ylimino) (IK-2014-006a) 1 HNMR-2-thiazolidine-2-ylimino)



Spectrum No. 9: Thiazolidin-2-(4-phenylthiazol-2-ylimone)-(2E,5E)-5-(dimethylamin) methylene (IK-2014-006a)

Mechanism of Formation of Compounds

1. IK-2014-004 compound training mechanism (a-b):

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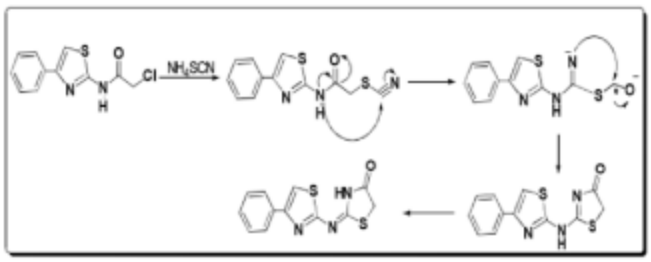
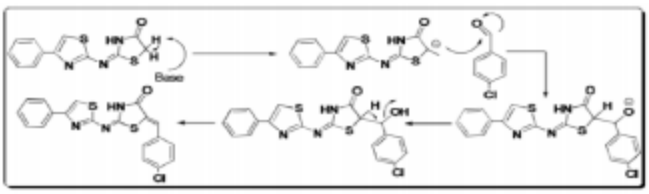
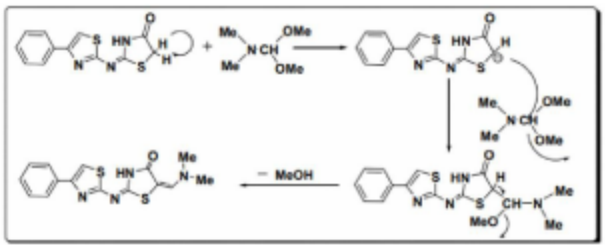


2. IK-2014-005 Compound Training Mechanism (a-h):



3. Mechanism of formation of compounds IK-2014-006(a-b):



**CONCLUSION-** The synthesis of thiazolyl 4-thiazolide-dimethylaminomethylene-methylene ketones aryl replaced explaining the synthesis of thiazolyl 4-thiazolidino-alpha, unsatutrated ketone and dimethylamino methylene ketones. 4-thiazolidinons aryl substituted IK-2014-004 are the primary compounds used for intermediate synthesization [IK-2014-005(a-b)] and [IK-2014-006(a-b)] (a-b). The composites [IK-2014-004(a-b)] were created in accordance with the schemes IK-2014-1 & IK-2014-2. The thiourea is replaced with phenacyl bromides (Scheme-IK-2014-1), which are converted into chloroacetamide [IK-2014-001(a-b)], are cycled to form the corresponding 2-amino thiazoles, with the effects of the chloroacetyl chloride compound [IK-2014-003 (a-b)] and with the cyclisation of ammonium thiocyanate, in reaction to obtain the appropriate 4-thiazolidinone derivatives Accordingly, (Scheme-IK-2014-1).

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