



Syntheses and Mass Spectra Investigation of Coumarino – [4,3-d] – Pyrimidine Derivatives

*Jehan A. Hasanen

* Chemistry department, faculty of science, suez canal university, ismailia, Egypt

ABSTRACT

Potassium salt of 2 mercapto – 4 – hydroxy – 5H [1] – benzopyrano – [4,3-d] – pyrimidin – 5 – one (2) was prepared by reaction of 1 with thiourea in presence of anhydrous potassium carbonate. Treatment of 2 with substituted phenacyl bromides , hydrazine hydrate and ammonia yielded the corresponding 2-(substituted) thio – 4 hydroxy – benzopyrano – pyrimidines (3a,b), 2-hydrazino- 4- hydroxyl- 5H-[1]- benzopyrano- [4,3-d]- pyrimidin- 5-one (4) and 2- amino-4-hydroxy- 5H-[1]-benzopyrano- [4,3-d]pyrimidin- 5-one (6), respectively.

Acetylation of 6 with acetic anhydride gave the corresponding *n*- acetyl derivative (7). 2-(substituted) amino – 4 – hydroxy -5H – [1] – benzopyrano – [4,3-d] – pyrimidin-5-one (8) was prepared via the reaction of 6 with substituted phenacyl bromides . The electron impact mass spectra of both the above compounds have also been recorded and their fragmentation pattern is discussed.

Keywords :

Introduction

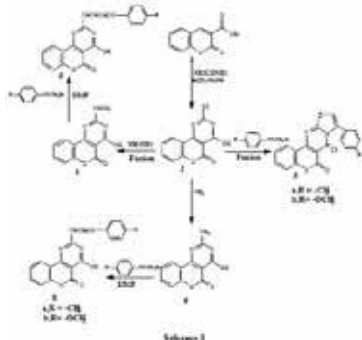
In the courses of recent investigation (1-6) involving 3-ethoxy carbonyl coumarin and thiourea in presence of anhydrous potassium carbonate , it was found that potassium salt of 2-mercapto -4- hydroxy-5H-[1] benzopyrano-[4,3-d] – pyrimidin-5-one (2) is converted into 2-hydrazino – (or 2 – amino –) – 4-hydroxy -5H-[1]-benzopyrano –[4,3-d]pyrimidin-5-ones (4 and 6) by the action of hydrazine hydrate and ammonia under fusion. The fact that only limited information is available on the mass spectra of 2- substituted- 4- hydroxyl- 5H-[1]benzopyrano-[4,3-d]-pyrimidin-5-ones (4 and 6), along with the preparation of fused coumarino –[4,3-d] – pyrimidin –5-ones .has prompted us to report their syntheses and study their electron impact (EI) mass spectral fragmentation .

Results and discussion

Chemistry

The potassium salt of 2-mercapto -4-hydroxy- 5H-[1]- benzopyrano –[4,3-d] – pyrimidin –5-one (2) was prepared from the reaction of 3- carboethoxy coumarin (1) with thiourea in the presence of anhydrous potassium carbonate in methanol under reflux.

Heating the potassium salt of 2-mercapto -4-hydroxy -5H-[1]-benzopyrano –[4,3-d] – pyrimidin –5-one (2) with alkyl halides (such as 4-methylphenacyl bromide and 4-methoxyphenacyl bromide) under fusion conditions gave the corresponding 3-(*p*-substituted phenyl)-[1] – benzopyrano[4,3-d] – thiazolo[2,3-b]-pyrimidin –4,5-diones (3a,b).



Hydrozoinolysis of potassium salt of compound 2 with hydrazine hydrate by fusion at 130°C, gave the corresponding 2-hydrazino -4-hydroxy-5H-[1] – benzopyrano-[4,3-d]-pyrimidin –5-one (4) .

Treatment of compound 4 with alkyl halides (namely 4-methyl phenacyl bromide and 4-methoxyphenacyl bromide) in dimethyl formamide under reflux yielded the corresponding 2- alkyl hydrazine- 4- hydroxyl -5H-[1] – benzopyrano –[4,3-d]- pyrimidin-5-ones (5a,b).

Amonolysis of potassium salt of 2-mercapto-4- hydroxyl -5H – [1]- benzopyrano - [4,3-d] – pyrimidin –5-one (2) with ammonia from ammonium acetate and/or formamide under fusion afforded the corresponding 2-amino -4-hydroxy – 5H –[1] –benzopyrano –[4,3-d] – pyrimidin –5-one (6, scheme 1) Acetylation 7 of compound 6 with acetic anhydride under reflux yielded the corresponding 2- acetylamino- 4-hydroxy -5H –[1] –benzopyrano – [4,3-d] – pyrimidin – 5 – one (7) The reaction of 2 amino – 4-hydroxy -5H –[1] – benzopyrano –[4,3-d] – pyrimidin – 5-one (6) with alkyl halides (such as 4- methylphenacyl bromide and 4 – methoxyphenacyl bromide) in dimethyl formamide under reflux produced 2- (substituted) amino – 4- hydroxyl -5H-[1]-benzopyrano –[4,3-d] – pyrimidin – 5- ones (8 a , b ; scheme 1) .

Mass spectrometry

The mass spectral decomposition modes of 8, 9 of some fused coumarino – [4, 3-d] – pyrimidin derivatives contaning different substituents in position two have been investigated and fragmentation pathways have been suggested.

Compounds 5a, b

The mass spectra of compounds 5a,b figure (1, 2) are fully consistent with the assigned structures.

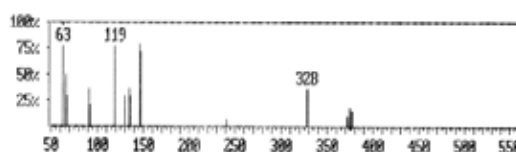


Fig. 1

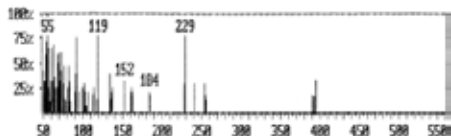
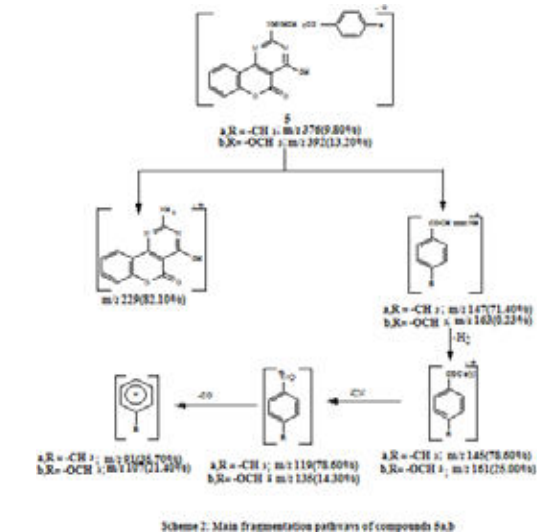


Fig. 2



Scheme 2: Main fragmentation pathways of compounds 5a,b

Thus, compounds 5a, b showed intense molecular ion peaks at m/z 376 and 392, consistent with the molecular formula $C_{20}H_{16}N_4O_4$ and $C_{20}H_{16}N_4O_5$, respectively.

The molecular ion of compounds 5a and 5b (scheme 2) underwent fragmentations to produce peaks at m/z 147 and m/z 163. It further underwent loss of H_2 , CN and CO to give peaks at m/z 145, 161, m/z 119, 135 and m/z 91, 107 respectively. Base peaks at m/z 63 and 55, found in the MS of compounds 5a and 56. The molecular ions of compounds 5a and 56 were also found to undergo fragmentations to produce the ion of 2-amino-4-hydroxy-5H-[1] benzopyrano-[4,3-d] pyrimidin-5H-[1] benzopyrano-[4,3-d]-pyrimidin-5-one at m/z 229.

Compounds 6 and 7

The mass spectrum of compounds 6 and 7 showed an intense molecular ion peaks at m/z 229 and m/z 271, corresponding to the molecular formula $C_{11}H_7N_3O_4$ and $C_{13}H_9N_3O_4$, respectively. The molecular ion of compound 6 (figure 3) underwent fragmentation to produce a peak at m/z 214 by losing NH group. The loss of cyano group (CN) from the ion at m/z 214 resulted in an ion at m/z 188. The ion at m/z 188 underwent loss of CO and NH_2 to give peaks at m/z 160 and 144, respectively.

Also, the molecular ion at m/z 229 underwent loss of amino group (NH_2) to give peak at m/z 213, which further broke to give an ion at m/z 187.

The ion of m/z 187 broke to give an ion at m/z 173 which lost nitrogen atom. Ion of m/z 173 fragmented to give ion of m/z 145 which lost carbonyl group (CO).

The molecular ion of compound 7 (m/z 271, figure 4) had fragmented to give the stable fragmented ion of m/z 229, corresponding to the molecular ion of compound 6 by losing $CH_2=C=O$ ketene molecule. The stable fragmentation of m/z 229 was broken via pathway in the same fragmented processes which was observed for compound 6 (scheme 3).

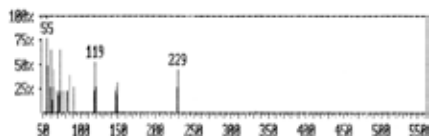


Fig. 3

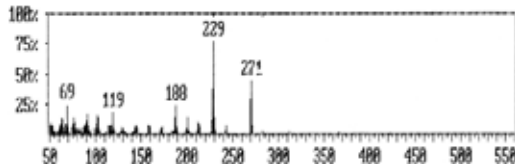
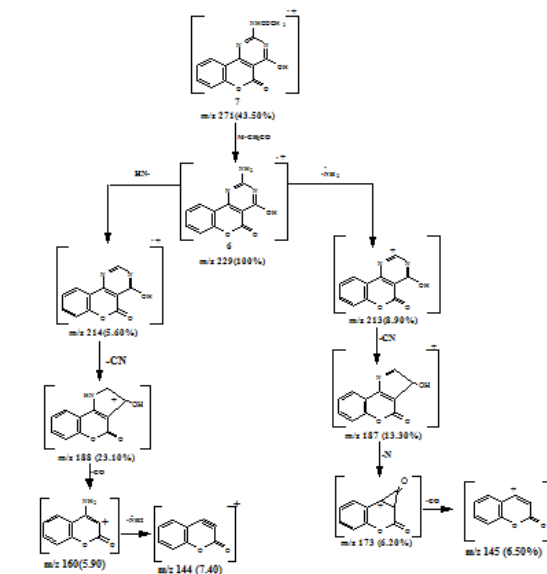


Fig. 4



Scheme 3: Main fragmentation pathway of compounds 6 and 7.

Compounds 8a and 8b

The molecular ion peaks of compounds 8a and 8b (figure 5 and 6) were observed at m/z 361 and m/z 377, corresponding to the molecular formula $C_{20}H_{11}N_3O_4$ and $C_{20}H_{11}N_3O_5$, respectively.

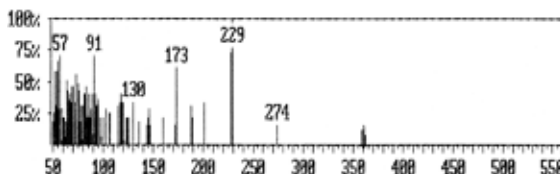


Fig. 5

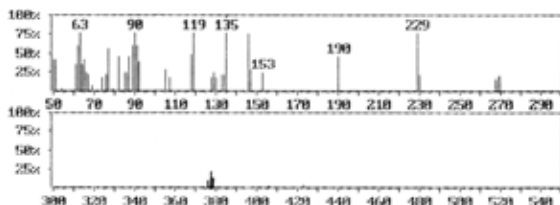


Fig. 6

The molecular ion of compounds 8a and 8b underwent fragmentations to produce stable peaks at m/z 229, corresponding to the molecular ion of compound 6. The fragmentation of m/z 229 which has further broken via pathway similar to compound 6.

Also, the molecular ion peaks at m/z 361 and m/z 377 underwent loss of 4-amino-4-hydroxy-5H-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one to give peaks at m/z 119 and m/z 135. It further underwent loss of carbonyl monoxide (CO) to give peaks at m/z 91 and m/z 107. The electron impact ionization of compound 8a it was found

that the base peak at m/z 229, while the base peak of compound 8b m/z 135.

Experimental

NMR Spectra Were recorded on a general electric QE300 instrument and chemical shifts were given with respect to TMS. IR spectra were recorded on a perkin- Elmer 1420 spectrometer and a biorld FTS7 (KBr). Mass spectra were recorded on a V G Autspec (EI and FAB+) and Hewlett Packed Ms-Engin Therom spray and ionization by electron impact at 70 eV. The accelerating voltage was 6 KV , the temperature of the ion source was ~ 200oC and the emission current was ~ 100 mA. Microanalyses were conducted using an elemental analyzer 1106. Melting points were determined on a Reichert Hot stage and were uncorrected Potassium salt of 2-mercapto - 4- hydroxyl-5H-[1]-benzopyrano-[4,3-d]-pyrimidin -5-one (2) A mixture of 1 (0.01mol), thiourea (0.01mol), and anhydrous potassium carbonate (0.03 mol) in methanol (50 ml) was heated under reflux with stirring for 2hr. The solid formed upon hot was filtered off and dried to give 2 as pale yellow powder, yield 63%.

3-(p-substituted phenyle) , benzopyrano-[4,3-d] – thiazolo – [2,3-b] – pyrimidin- 4,5-diones (3a,b) A mixture of 2 (0.01 mol) and alkyl halides (namely . 4-methylphenacyl bromide and 4-methoxy phenacyl bromide) (0.01 mol) was fused in an oil bath at 130oC for 2 H and then was cooled and poured into water . The solid obtained was filtered off , washed with water , dried and purified by recrystallization (ethanol) to give 3.

3-(p_methylphenyle) –[1]- benzopyrano-[4,3-d] – thiazolo-[2,3-d] pyrimidin -4,5- dione (3a) as orange crystals , yield 67 % , m.p. 95oC IR (KBr) : 1743 , 1695 (C=O),1625 (C=N) , 1605,1585 (C=C) , 1110, 1072 (C-O) cm-1. δ H (DMSO-d6) : 2.35 (S, 3H,CH3) , 7.13-8.12 (m,9H, Ar-H and thiazol-H) ppm. MS: m/z (%) = 362 (M++2, 3.60), 348(3.10) , 345(8.70) , 344(8.20), 343 (7.20), 336(3.10), 335(4.10), 333(3.60), 331(4.10) , 322(4.10) , 320(3.60) , 309(4.60) , 305(6.70) , 304(6.70) , 293(5.10) , 292 (4.10) , 277(7.20) , 276(3.60) , 275(4.10) , 261(5.60) , 250(6.70) , 249(4.60) , 241(3.60) , 234(5.60) , 233(6.70) , 229(4.10) , 222(6.20) , 221(8.20) , 217(6.20) , 216(5.60) 204(5.60) , 203(7.70) , 202(5.60) , 192(3.10) , 190(15.90) , 189(6.20) , 188(6.70) , 178(5.10) , 175(6.70) , 174(6.70) , 166(3.60) , 163(5.10) , 161(4.60) , 160(6.70) , 159(7.70) , 148(9.70) , 147(18.50) , 140(3.60) , 139(7.20) , 138(3.60) , 135(3.60) , 134(11.80) , 133(6.70) , 120(6.20) , 119(100) , 118(23.60) 117(8.70) 115(8.20) 114(8.70) , 103(6.70) , 102(5.60) , 96(7.70) , 91(46.70) , 79(4.60) , 77(4.60) 76(10.30) , 65(21.00) , 64(16.40) , 63(5.10).

Anal. C₂₀H₁₂N₂O₃S for calcd : C , 66.66 ; H,3.33; N, 7.77; N,8.88. Found : C,66.36 ; H, 3.13 ; N,7.47 ; S,8.44.

3-(p-methoxyphenyl) – [1] – benzopyrano [4,3-d] – thiazolo-[2,3-b] – pyrimidin-4,5-dione(3b) as yellow crystals, yield 63% , m.p. 98OC . IR (KBr) : 1742 , 1689 (C=O) , 1623(C=N), 16.5, 1589 (C=C) , 1120 , 1083 , 1030 (C-O) cm -1 . δ H (DMSO-d6) : 3.83 (S,3H,OCH3) , 7.12-8.12 (m,9H, ArH and thiazole -H) ppm. Ms : m/z (%) = 377 (m++1,1.30) , 376(M+2.30) , 361(33.30) , 344(27-80) , 343(38.90) , 293(27.80) , 206(33.30) , 190(50.00) , 148(33.30) , 147(5.60) , 146(38.90) , 145(33.30) , 137(22.20) , 136(50.00) 122(33.30) , 121(33.30) , 120(33.30) , 119(83.30) , 118(50.00) , 117(66.70) , 116(22.20) , 93(27.80) , 91(100) , 90(27.20) , 89(88.90) 88(83.30) , 83(27.80) , 82(44.40) , 81(88.90) 80(55.60) , 79(66.70) . 78(50.00) , 77(5.60) , 75(5.60) 71(33.30) , 69(50.00) , 66(22.20) , 65(50.00) , 64(16.70) , 62(16.70) , 60(38.90) , 59(11.10) , 57(16.70) , 54(44.40) , 52(55.60) 50(11.10) , Anal. C₂₀H₁₂N₂O₄S for Calcd : C, 63.83 ; H,3.19 ; N, 7.44 ; S,8.51 . Found :C, 63.63 ; H,3.03 ; N,7.23 ; S,8.31.

2-Hydrazino -4-hydroxy-5H-[1]-benzopyrano-[4,3-d]-pyrimidin-5-one(4).

A solution of 2 (0.01 mol) in hydrazine hydrate (15 ml) was heated under reflux at 130OC for 25-30 min . The solid formed on hot was filtered off and washed with ethanol , dried and purified by recrystallization with dimethyl formamide to give 4

as pale orange crystals , yield 71% , m.p . 360OC. IR (KBr): 3430-2560(br.oH), 3336, 3175(NH₂), 3251(NH), 1718(C=O), 1616(C=N) , 16.3, 1589(C=C) , 1125, 1045(C-O) cm -1 . δ H(DMSO-d6) : 4.36(S,2H,NH₂) , 7.29—7.78(m,4H,,Ar-H) , 8.83 (br.S, 1H , OH) ,9.35(S,1H,NH)ppm. MS : m/z(%) = 246(M++2,11.5) , 245(M++1,11.20) , 244(100) 230(23.50) , 229(25.80) , 228(10.60) , 216(11.20) , 215(69.70) , 214(16.90) 213(16.90) , 197(13.70) , 188(26.40) , 187(7.70) , 186(11.50) , 160(7.90) , 158(12.80) , 145(9.80) , 144(5.60) , 130(7.00) , 129(7.00) , 126(8.60) , 119(18.60) , 116(15.00) 103(23.40) , 102(24.00) , 89(20.60) , 88(20.90) , 87(10.30) , 92(19.50) , 91(26.60) , 90(17.00) , 77(18.70) 76(24.30) , 51(16.40) . Anal C₁₁H₈N₄O₃ for Calcd : C, 54.10; H, 3.30; N,22.93. Found : C 53.97;H,3.19;N,22.71.

2- Alkylhydrazino-4- hydroxyl -5H-[1]-benzopyrano-[4,3d]-pyrimidin-5-ones(5a,b).

A mixture of 4 (0.01 mol) and a reagent such as 4-methyl bromide and 4- methoxy phenacyl bromide (0.01mol) in dimethyl formamide (30 ml) was heated under reflux for 2hr , then cooled and poured into water . The deposited solid was filtered off , washed with water , dried and purified by recrystallization with ethanol to give 5.

2-(p-methylbenzoylmethyl) hydrazino- 4 – hydroxyl – 5H – [1] – benzopyrano –[4,3-d] – pyrimidin -5- one (5a) as yellow crystals , yield 65% , m.p. 205OC . IR (KBr) : 3225 (NH) , 3401-2820(br.OH), 1741 , 1703 (C=O) , 1623 (C=N) , 16.5,1589 (C=C),1125, 1081 (C-O) cm-1 . δ H (DMSO-d6) : 2.41 (S,3H,CH₃) < 3.81 (S,2H,NHCH₂CO) , 7.1-7.81(m,8H,Ar-H), 8.31(br.S,1H,OH), 9.02(S,1H,NH) , 11.10(S,1H,NH)ppm. MS: M/z (%) = 377 (M++1,2.3) , 376(M+9.80) , 329(35.70) , 328(35.70) , 241(7.10) , 147(71.40) , 146(78.60) , 136(28.60) , 135(35.70) , 130(28.60) , 119(78.60) , 92(21.40) , 91(35.70) , 67(28.60) , 66(50.00) , 63(100) .

Anal. C₂₀H₁₆N₄O₄ For Calcd: C, 63.83; H, 4.25; N, 14.89. Found : C,63.63;H,14.69.

2-(4-methoxybenzoylmethyl) hydrazine – 4 – hydroxy -5H-[1]-benzopyrano-[4,3-d] – pyrimidin -5-one(5b) as yellow crystals , yield 63% , m.p. 240OC . IR(KBr) : 3390-2850(br-oH), 3287(NH), 1741, 1703 (C=O) , 1622(C=N), 1606, 1589(C=C) , 1125, 1081, 1030 (C-O) Cm -1 .

δ H(DMSO-d6):3.61(S,3H,OCH₃),3.85(S,2H,NHCH₂CO),7.13-7.81(m,8H,Ar-H),8.31(br.S,1H,OH), 9.10(S,1H,NH) ,11.02 (S,1H,NH)ppm.MS: m/z(%)= 393(M++1,11.20), 392(M+13.20) , 256(14.30) , 255(17.90) , 254(28.60), 241(28.60) , 229(82.10) , 228(28.60) , 185(17.90) , 184(21.40) , 162(21.40) , 161(25.00) , 160(21.40) , 152(32.10) , 137(25.00) , 136(21.40) , 135(14.30) , 134(39.30) , 119(78.60) , 118(14.30) , 144(25.00) , 113(17.90) , 107(21.40) , 103(21.40) , 102(28.60) , 101(25.00) , 99(25.50) , 95(21.40) , 92(75.00) , 91(39.30) , 90(21.40) , 83(32.10) , 77(14.80) , 76(46.40) , 75(28.60) , 73(60.70) , 65(25.00) , 64(35.70) , 63(67.90) , 56(64.30) , 55(100) , 54(71.40) , 53(57.10) , 51(32.10) Anal. C₂₁H₁₆N₄O₅ for Calcd : C, 61.22; H,4.08; N,14.28. Found: C, 61.01; H, 3.98; N, 14.03.

2- Amino- 4 – hydroxyl-5H-[1]- benzopyrano [4,3-d] – pyrimidin-5-one (6).

A mixture of 2 (0.01) and ammonium acetate (0.05 mol) or formamide (15 ml) was fused on oil- bath at 130OC for 2hr, then cooled and poured into water. The solid obtained was filtered off, washed with water, dried and purified by recrystallization with butanol to give 6 as pale yellow crystals, m.p. 386 C. IR(KBr) : 3405-2810(br.oH), 3312,3185(NH₂) , 1725(C-O) , 1628(C=N), 1603,1591(C=C), 1213, 1085(C-O) cm -1 . δ H(DMSO-d6): 5.21(S,2H,NH₂) , 7.21-7.78(m,4H,ArH), 8.53(br.S,1H,OH) ppon. MS:m/z(%) = 229(M+43.50), 228(M+1,26.10) , 149(30.40) , 148(17.40) , 147(26.10) , 146(21.70) , 120(26.10) , 119(52.20) , 118(21.70) 91(26.10) , 85(39.10) , 82(21.70) , 81(21.70) , 76(21.70) , 73(65.20),

72(21.70), 71(17.40), 69(21.70), 64(43.50), 63(26.10), 62(13.00), 60(65.20), 59(26.10), 56(47.8), 55(100).

Anal. C₁₁H₇N₃O₃ for calcd: C, 57.64; H, 3.06, N, 18.34. Found: c, 57.46; H, 2.96; N, 18.3.

2-(acetyl)amino-4-hydroxy-5H-[1]- benzopyrano-[4,3-d]-pyrimidin-4-one(7).

A solution of 6 (0.01 mol) in acetic anhydride (20ml) was heated under reflux for 2hr. then cooled and poured onto crushed ice-water. The product formed was collected by filtration , washed with water , dried and crystallized (ethanol) to give 7 as yellow crystals , yield 67% , m.p. 285 C.

IR(KBr) : 3380-2851(br.oH), 3225(NH), 1728,1705(C=O), 1621(C=N), 1607,1588(C=C), 1210, 1083 (C-O) cm⁻¹ δH (DMSO-d₆): 2.51(S,3H,COCH₃), 7.12-7.28(m,4H,Ar-H), 8.74(S,1H,OH),12.10(S,1H,NH) ppm . MS: m/z (%) = 272(M++1,7.40), 271(M+,43.50), 230(14.20),229(100), 228(37.60), 227(10.90), 214(5.60), 213(8.90), 212(9.20), 201(13.30), 189(5.90), 188(23.10), 187(13.30), 186(3.60), 173(6.20), 172(3.80), 160(5.90), 159(6.50) 158(6.50), 146(3.80), 145(6.50), 144(7.40), 143(3.80), 131(3.60) 130(4.40) 129(5.90), 120(3.80), 119(18.30), 118(7.70), 117(2.70), 116(6.50), 115(7.40), 114(6.80), 104(4.70), 103(13.60), 102(14.8), 101(8.30), 91(17.20), 90(10.90), 89(8.00), 77(9.20), 76(13.60), 75(8.60), 69(24.00), 68(8.90), 65(6.20), 64(12.40), 63(13.30), 53(7.70), 52(6.80), 51(10.40), 50(7.70) .

Anal. C₁₃H₉N₃O₄ for Calcd : C, 57.56; H,3.32; N,15.49 . Found : C, 57.33 ; H, 3.17; N, 15.28.

2- (alkyl) amino- 4 – hydroxyl – 5H – [1] – benzopyrano- [4,3-d] – pyrimidin- 5- ones (8a,b).

A mixture of 6 (0.01 mol) and alkyl halides (namely , 4 –methyl phenacyl bromide and 4-methoxy phenacyl bromide) (0.01 mol) in dimethyl formamide (30ml) was heated under reflux for 3hr . The solid obtained after cooling was filtered off , washed with water , dried and recrystallized (ethanol) to give 8.

2- (p-methulobenzoylmethyl) amino- 4- hydroxyl -5H-[1] benzopyrano –[4,3-d] – pyrimidin – 5-one(8a) as pale yellow crystals , yield 68% , m.p : 210 OC. IR(KBr) : 3390-2810 (br.OH), 3223(NH), 1725, 1689(C=O), 16199(C=N), 1605,1588(C=C), 1210, 1087(C-O) cm⁻¹ δH(DMSO-d₆) : 2.34(S,3H,), 3.58(S,2HNCH₂CO), 7.12-7.83(m,8H,ArH), 8.73(S,1H,OH), 10.35(S,1H,NH) ppm .

MS: m/z(%) = 362(M++1, 2.30), 361(M+,13.20), 229(100), 228(72.70), 201(33.30), 189(21.20), 188(30.30), 173(60.60), 172(15.20), 160(21.20), 147(15.20), 146(27.30), 145(21.20), 144(15.20), 136(18.20), 130(33.30), 125(21.20), 124(21.20), 123(21.20), 120(33.30), 119(39.40), 118(39.40), 117(33.30), 115(30.30), 107(24.20), 106(24.20), 103(27.30), 100(21.20), 97(21.20), 95(36.40), 92(39.40), 91(69.70), 90(9.10), 89(39.40), 88(27.30), 86(39.40), 83(45.50), 82(39.40), 77(18.20), 76(42.20), 75(48.50), 73(54.50), 72(33.30), 70(45.50), 69(45.50), 65(42.40), 64(51.50), 63(18.20), 57(69.70), 55(66.70), 54(57.60), 52(57.60), 51(24.20). Anal. C₂₀H₁₅N₃O₄ for Calcd : C,66.48; H, 4.15 ; N,11.63. Found : C,66.24 ; H, 4.03 ; N, 11.52 .

2- (4- methoxy benzoylmethyl) amino – 4 – hydroxyl –H- [1] benzopyrano –[4,3-d] – pyrimidin-5-one (8b) as yellow crystals , yield 69 % , m.p : 145OC . IR (KBr): 3398-2950(br-OH), 3229(NH), 1725, 1693(C=O), 1623(C=O), 1610, 1589(C=C), 1210,1085,1035(C-O) cm⁻¹ . δH(DMSO-d₆) : 3.63 (S,2H,NHCH₂CO), 3.85(S,3H,OCH₃), 7.12-7.83(m,8H,ArH), 8.71(S,1H,OH), 10.36(S,1H,NH) ppm. MS: m/z (%) = 378(M++1,1.70), 377(3.20), 230(20.70), 229(79.30), 190(44.80), 153(24.10), 147(27.60), 146(75.90), 135(100), 134(20.70), 133(20.70), 130(17.20), 129(24.10), 128(17.20), 119(89.70), 118(48.30), 107(17.20), 105(27.60), 92(37.90), 91(58.60), 90(82.80), 89(58.60), 87(44.80), 86(24.10), 85(24.10), 82(44.80), 77(55.20), 76(20.70), 74(17.20), 67(20.70), 66(24.10), 65(41.40), 64(34.50), 63(79.30), 62(58.60), 61(34.50), 51(41.40), 50(41.40).

Anal. C₂₀H₁₅N₃O₅ for Calcd : C,63.66; H,3.97 ; N,11.14. Found : C,63.43 ; H, 3.78; N, 11.02.

REFERENCES

- 1-I. el-deen, H.k.ibrahim , Phosphours , Sulfer , Silicon Relat , 179, 195 (2000). | 2- I. M. el-deen, H.k.ibrahim , Phosphours , Sulfer , Silicon Relat , 160, 241 (2000). | 3- I. M. el-deen, H.k.ibrahim , Chem. Pap. , 58, 200 (2004) | 4- I. M. el-deen, H.k.ibrahim, F. F. Mohamed, Chin. J. Chem., 18, 590 (2000). | 5- S.M.Mohamed, Bull. Chem. Technol. Macedonia, 24, 117 60 (2005). | 6- H.K.Ibrahim , J.A.Hassanen , Afinidad , 64 (527) , 60 (2007). | 7- H.K.Ibrahim , J.A.Hassanen ,M.A.Zein , I.M-El-Deen , Mens Agitat , 3 (1) , 59 (2008) . | 8- Shivakama Holla B.,Shivananda M.K.,Akberail,P.M., J.Indian Chem.Soc.,75,532 (1998). | 9- I.M.El-Deen, M-E.abd El-Fattah, Bull . korean Chem.Soc. , 24, 47 (2003).