ıdian J. Phys. **79** (3), 297-299 (2005)

Note

INTERNATIONAL JOURNAL
IJP

Synthesis and characterization of two derivatives of 2-mercapto-3-m-tolyl-3H-quinazolin-4-one

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Received 25 May 2004, accepted 20 January 2005

ıstract : Quinazoline derivatives are of special importance because of their versatile biological and pharmacological activities The 2-(2-oxo-ıpylsulfanyl)-3-m-tolyl-3H-quinazolin-4-one(QA) and 2-(4-oxo-3-m-tolyl-3,4-dihydro-quinazo- lin-2-ylsulfanyl)-propionic acid methyl ester(QP) re synthesized by thiolating 2-thioxo-3-m-tolyl-2,3-dihydro-1H-quinazolin-4-one by an easy and efficient microwave-assisted method The structures the compounds have been confirmed on the basis of their IR, PMR spectral and elemental analyses Further, characterization for crystal parameters l line broadening effects have been done.

ywords · Quinazolinone, MW alkylation , X-ray diffraction

.CS Nos. 61.10.Nz, 78.30.Jw

ınazoline derivatives are of special importance because of ɜır versatile biological and pharmacological activities [1-6] pecially anti-inflammatory, anti-convulsant, hypnotic, thelmintic, hypotensive, antibacterial *etc* Avoiding organic lvent, microwave irradiation in organic reactions is used for ort reaction time, operational simplicity and formation of ɜaner reaction products. The use of thermal energy source in rious organic reactions [7] is common and the use of domestic icrowave oven in this regard is now a well-established ocedure in microwave organic reaction enhancement (MORE) l chemistry to obtain S-alkylated derivatives of thioquinazoline ıng different alkylating agents. Alkylation using catalytic ıount of dimethyl formamide (DMF), the polar molecule, ıproves energy transfer and allows higher temperatures [9, 10] d less time compared to the conventional method of S-kylation which requires a longer reaction time at a regulated nperature [11].

All m.p's. were determined in open capillary tubes and are ıcorrected. IR spectra were recorded in KBr on Shimadzu IR-7 spectrophotometer and PMR spectra in CDCl3 and DMSO-ı on Perkin-Elmer R-32 spectrometer using Trimethylsilane as an internal standard. Thin layer chromatographic plates coated with silica-gel-G were used to check the purity of the compounds. Microwave irradiation was carried out in the domestic SHARP microwave oven. The X-ray diffractograms were recorded on Philips PW 3710 diffractometer with scanning rate 2°/ min. attached to a digital computer along with graphical assembly of Cuk_α radiation source observed at Cu-Ni 40 kV / 40 mA.

2-(2-Oxo-propylsulfanyl)-3-m-tolyl-3H-quinazolin-4-one (QA):

2.68 g (10 mmol) of 2-thioxo-3-m-tolyl-2,3-dihydro-1H-quinazolin-4-one, 1.84 g (20 mmol) of chloroacetone and 0.5 ml of DMF and powdered NaOH (0.5 g), smoothly mixed and placed inside a Pyrex-glass and the mixture was irradiated with microwaves (900 W /2450 MHz frequency) for 8 minutes, the mixture was then added with 2N HCl (10 ml). The solid separated was filtered, conveniently dried and recrystallized from ethanol to get 2.27g of (QA), yield 69.5%, mp. 142°C, IR(KBr): 1725(C = O), 1675(cyclic C=O), 1620 cm^{-1}(C=N), PMR (DMSO-d_6): 2.4(3H,s,-$COCH_3$), 3.6-4.0(2H,s, -SCH_2), 7.2-8.2 (8H,m,Ar-H). Anal. Calcd for $C_{18}H_{18}N_2O_2S$: C, 66.23; H, 5.56; N, 8.58 %; Found: C, 66.2; H, 5.55; N, 8.6 %.

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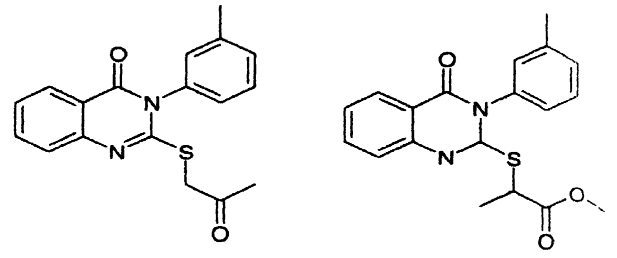
2-(4-oxo-3-m-tolyl-3,4-dihydro-quinazolin- 2 - ylsulfanyl) - propionic acid methyl ester (QP) :

2.68 g (10 mmol) of 2-thioxo-3-m-tolyl-2,3-dihydro-1H-quinazolin-4-one, 2.44 g(20 mmol) of methyl 2-chloropropionate and 0.5 ml of DMF and powdered NaOH (0.5 g), were smoothly mixed and placed inside a Pyrex-glass and the mixture was irradiated with microwaves (900 W /2450 MHz frequency) for 5 minutes, the mixture was added with 2N HCl (10 ml). The solid separated was filtered, conveniently dried and recrystallized from ethanol to get 2.67g of (QP), yield 75.2%, mp. 145°C, IR (KBr): 1755(C=O), 1710 (cyclic C=O), 1620(C=N), 1060 cm^{-1} (-O-). PMR (DMSO-d_6): 1.4(3H, d, J=7Hz., CH_3), 2.35(3H, s, Ar-CH_3), 3.75(3H, s, OCH_3), 4.3(1H, q, J=7.5Hz., S-CH), 7.2-8.2 (8H, m, Ar-H) Anal. Calcd for $C_{19}H_{20}N_2O_3S$: C, 64.02; H, 5.66; N, 7.86 %; Found: C, 64.0; H, 5.64; N, 7.9 %.

The proposed structures of (QA) and (QP) are presented in Figure 1 on the basis of IR, PMR spectral and elemental analyses.

The X-ray diffraction pattern of (QA) and (QP) with respect to their prominent peaks have been indexed using computer trial and error method [12], for a good fit between observed and calculated *d* values. The relative intensities corresponding to the prominent peaks have been calculated by normalizing ther with respect to their maxima.



(QA) (QP)

Figure 1. Structures of (QA) and (QP).

The indexing of diffractogram of (QA) and (QP) are presente in Table 1. The indexing of diffractogram of both the compound are nearly identical and it can be proposed that the compound belong to same structural class. The comparison of the value 'd' reveals that there is good agreement between the calculate and observed value of 'd' on the basis of assumption orthorhombic crystal structure [13-16]. The compound (QA) ha

Table 1. X-ray diffraction data of 2-(2-oxo-propylsulfanyl)-3-m-tolyl-3H- quinazolin-4-one (QA) and 2-(4-oxo- 3-m-tolyl-3, 4-dihydro- quinazolin 2-ylsulfanyl)-propionic acid methyl ester (QP)

QA						QP					
Peak No.	*2θ deg.*	d_{obs}	d_{cal}	*h k l*	*RI %*	*Peak No.*	*2θ deg*	d_{obs}	d_{cal}	*h k l*	*RI %*
1	13.180	9.975	9.975	0 2 0	10.4	1	11.600	11.328	11.328	0 1 0	10.5
2	13.920	9.447	9.447	1 1 0	100	2	12.690	10.359	10.359	1 0 0	100
3	14.235	9.239	9.239	0 0 1	8.4	3	14.410	9.128	9.128	0 1 1	21.9
4	18.645	7.067	7.001	1 0 1	28.2	4	19.305	6.827	6.848	1 1 1	12.4
5	19.680	6.699	6.651	0 3 0	24.1	5	23.445	5.634	5.664	0 2 0	41.2
6	20.200	6.528	6.605	1 1 1	5.0	6	26.295	5.033	4.970	1 2 0	6.2
7	24.665	5.360	5.363	2 0 0	3.4	7	29.215	4.539	4.564	0 2 2	32.7
8	26.815	4.937	4.987	0 4 0	23.4	8	29.745	4.460	4.504	2 1 1	25.3
9	27.445	4.826	4.822	1 3 1	7.0	9	30.355	4.372	4.299	2 0 2	9.4
10	28.050	4.724	4.723	2 2 0	10.2	10	31.455	4.223	4.264	1 1 3	12.0
11	29.070	4.561	4.523	1 4 0	3.6	11	35.475	3.757	3.776	0 3 0	47.3
12	29.595	4.482	4.500	0 1 2	14.5	12	37.680	3.545	3.547	1 3 0	18.8
13	30.360	4.372	4.389	0 4 1	4.5	13	40.490	3.308	3.303	3 1 0	6.5
14	35.185	3.787	3.794	0 3 2	16.2	14	42.990	3.124	3.151	3 0 2	6.8
15	36.030	3.701	3.739	1 5 0	10.4	15	45.140	2.982	2.982	2 1 4	6.5
16	36.415	3.664	3.663	0 5 1	17.7	16	46.465	2.920	2.919	1 3 3	3.4
17	36.545	3.651	3.652	2 4 0	19.6						
18	37.890	3.526	3.520	3 1 0	7.2						
19	38.810	3.445	3.447	2 1 2	5.8						
20	43.235	3.107	3.098	2 3 2	9.0						
21	44.590	3.017	3.019	0 5 2	5.8						

lattice constants $a = 10.7267$ Å, $b = 19.9514$ Å, $c = 9.2398$ Å and $V = 1977.43$ Å^3 and the compound (QP) has lattice constants $a = 10.359$ Å, $b = 11.328$ Å, $c = 15.412$ Å and $V = 1808.76$ Å^3, respectively.

The experimental density values of the (QA) and (QP) determined by specific gravity method, are found to be 0.9567 and 1.1459 g cm-3, respectively. The number of molecules (n) per unit cell was calculated ($\rho = nm/NV$) and are found to be 4. With this number theoretical density was fixed and are 1.0489 cm^{-3} for (QA) and 1.2576 g cm^{-3} for (QP), respectively. The other parameters such as pore fraction, packing fraction, thickness of particle and radius of molecule were calculated. By using experimental and theoretical density of (QA) and (QP), the pore fraction was computed and it comes out to be 39.48 and 31.76 %, respectively. The radius and the volume of molecules of (QA) and (QP) were calculated and are found to be 3.792 and 3.662 Å and 228.481 and 205.780 Å^3, respectively. The packing fraction were calculated using volume of molecule and volume of unit cell of (QA) and (QP) and are found to be 46.21 and 45.51%, respectively.

The particle size of the samples under study was determined using the equation $t = 0.9\lambda/B\cos\theta$ and are found to be 168 and 176 Å for (QA) and (QP), respectively. This parameter makes possible to distinguish between natural particle size and particle size due to broadening effect by calculating full width at half maximum (B) corresponding to its Bragg's θ. The plot of $B\cos\theta$ *versus* $\sin\theta$ was found to be straight line for both samples, parallel to X axis indicating the absence of any strains caused by inhomogeneous lattice distortions and compositional fluctuations. It is concluded that the compounds under investigation are homogeneous with respect to the particle size distribution.

Acknowledgments

Authors thank the In-charge, CFC, Shivaji University, Kolhapur for providing X-ray analysis facility.

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