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NITRATION OF 2,3- TRIMETHYLEN -3,4-DIHYDROQUINAZOLIN-4-ONES AND QUINAZOLINES

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Abstract: The nitration reactions of 2,3-polymethylene-3,4-dihydroquinazolin-4-one and quinazolines were studied . The reaction conditions for the substitution of hydrogen atoms in positions 6 and 8 of the aromatic ring were shown. It turned out that it is impossible to introduce a second nitro group into the quinazoline ring without using an excess amount of the nitrating mixture (1:4) and under relatively complex conditions (heating to 90-100 0C). The synthesized compounds were studied for the first time and it was found that such organic substances have biological activity. 2,3-Polymethylene-3,4-dihydroquinazolin-4-one was nitrated under special conditions to obtain several different nitro derivatives and their properties (reduction, substitution reactions) were studied. This nitration process was first carried out on three-ring quinazoline and quinazoline rings, and their properties were studied.

Key words:2,3-polymethylene-3,4-dihydroquinazolin-4-ones, ichinazolines , nitration, amination , transamination , enamine , acetone cyanohydrin , bromination .

VISIONS

Previously [1,3] we studied some electrophilic substitution reactions of 2,3-polymethylene-3,4-dihydroquinazolin-4 – ones . Some of the synthesized compounds have growth-regulating , hypnotic, muscle-relaxing , narcotic action. Therefore, they are of certain practical interest. On the other hand, the presence of several reaction centers (pyridine nitrogen atom, aromatic ring, etc.) make them interesting in chemical terms. Before our research, there are almost no data on nitration reactions of quinazalines in the literature . Therefore, the study of these reactions is of great scientific interest.

Purpose of the work:

To date, there are no data in the literature on the electrophilic substitution of 2,3-polymethylene-3,4-dihydroquinazolin-4-ones. There is only information on the nitration of the simplest representative of the quinazoline series - quinasolone-4 and some of its derivatives. [1] It was therefore of interest to investigate the nitration reactions of 2,3-polymethylene-3,4-dihydroquinazolin-4-ones and quinazolines .

For this purpose, we studied [2,3,4,5] the nitration of 2,3-polymethylene-3,4-dihydroquinazolin-4-ones (I-III). The reaction proceeds smoothly when I-III, IV are treated with a nitrating mixture (reagent ratio 1:2.3) and leads to 6-nitro-2,3-polymethylene-3,4-dihydroquinazolin-4-ones (V-XIII).

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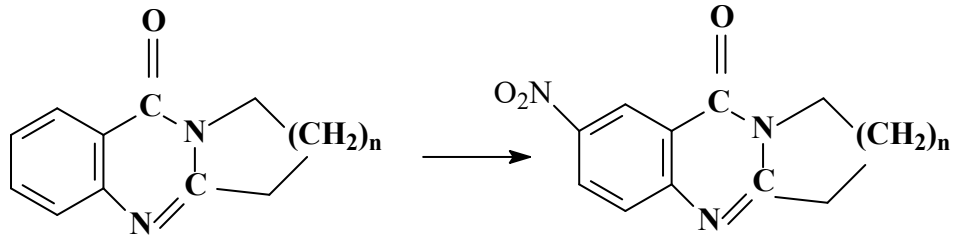
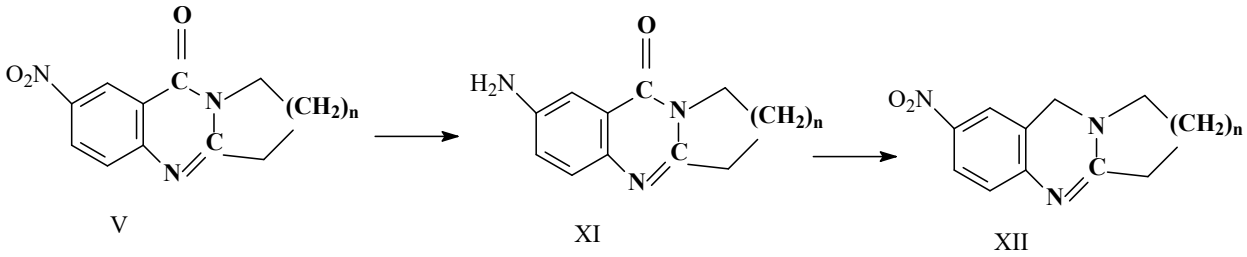


I-III, IV V-VIII

IV n= 1; IV -VI n=2; X, VII n=3; IV, VIII n=4

Compounds I-III, IV react with a mixture of nitric and sulfuric acids and give V-VIII under mild conditions. However, it is not possible to introduce a second nitro group into the quinazoline ring even when using an excess of the nitrating mixture (1:4.6) and under relatively harsh conditions (heating to 90-100 ^{0}).

The position of the nitro group of 6-nitro-2,3-polymethylene-3,4-dihydroquinazolin-4-ones has been proven by spectral data, their counter synthesis from 5- nitroanthranilic acid, and also by reduction with tin chloride to the corresponding 6-amino-2,3-polymethylene-3,4-dihydroquinazolin-4-ones [XI]. The latter are reduced with zinc in hydrochloric acid at room temperature and yield 6-amino-2,3-polymethylene-3,4-dihydroquinazolines XII.



V XI XII

The nitration reaction of 2,3-trimethylene-3,4-dihydroquinazoline (XIII) proceeds differently. When using a 1:1 reagent ratio, a mono nitro product is formed – 6-nitro-2,3-polymethylene-3,4-dihydroquinazoline (XIV), and at 1:2 – 6,8-dinitro-2,3-trimethylene-3,4-dihydroquinazoline (XV).

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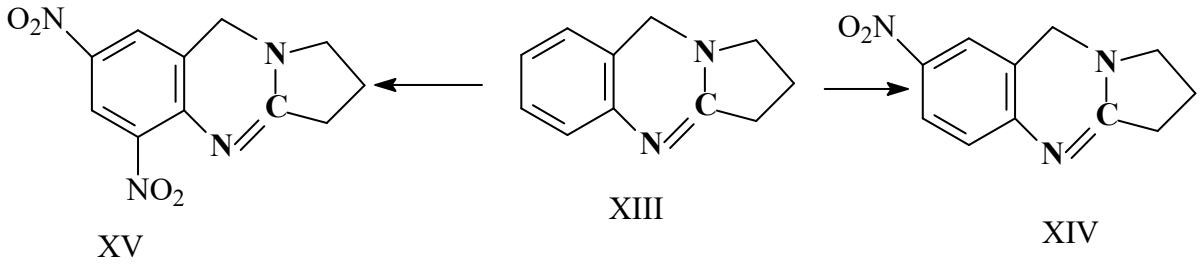


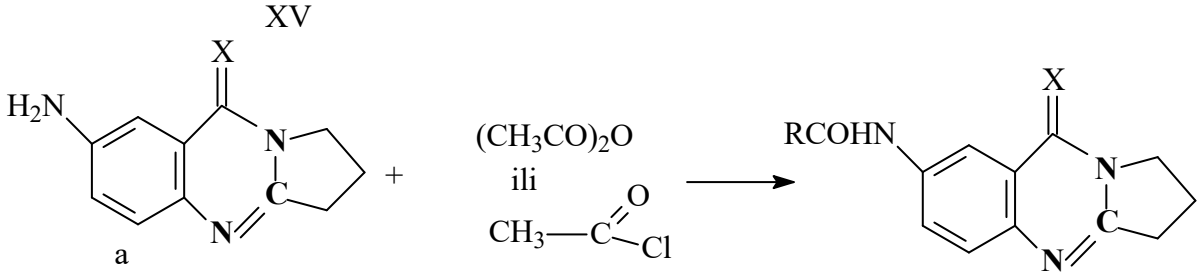
The possibility of introducing two nitro groups into the molecule of 2,3-trimethylene-3,4-dihydroquinazoline is apparently explained by an increase in nucleophilicity benzene ring during the transition from 2,3-trimethylene-3,4-dihydroquinazolone-4 to the corresponding quinazoline , which is associated with the withdrawal of electrons from the aromatic ring by the amide carbonyl .

Reduction of the nitro group of 6'-nitro-2,3-trimethylene-3,4-dihydroquinazoline gives 6'-amino-2,3-trimethylene-3,4-dihydroquinazoline, which is identical to the product obtained from 6'-nitro-2,3-trimethylene-3,4-dihydroquinazolin-4-one.

In the mass spectrum of XIV there is an intense peak of the molecular ion with m/e 262(M^+), 215(M^+-47), 182(M^+-80), 169(M^+-93), 149(M^+-113), 103(M^+-159), the relative intensity of which is 100, 25, 12, 54, 10, 9%, respectively.

In the PMR spectrum of compounds XIV and XV, the methylene protons at α-C, β-C, γ-C have chemical shifts at 2.80; 2.10; 3.50 and 2.98; 2.10; 3.63 ppm , respectively, and the protons of the methylene group at C-4 – at 4.55 and 4.73 ppm . The signals of the protons of the aromatic ring appear in the region of 6.80 ppm (doublet, J=8.0 Hz , H-8), 7.78 ppm (doublet-doublet, J_{ortho}=12 Hz , J_{meta}=2 Hz , H-7) and 8.70 ppm . (doublet, J_{meta} = 2.5 Hz , H-5) for XIV, and for XV – at 8.68 ppm (doublet, J = 2.5 Hz , H-5) and 7.98 ppm (doublet, J = 2 Hz , H-7). (**ppm-millionth of a decimal fraction**).

Acylation of 6-amino-2,3-trimethylene-3,4-dihydroquinazolin-4-one and -quinazoline with acetic anhydride or methyl chlorocarbonate leads to the corresponding acyl derivatives (XVI a, b).



a) R=CH_3, X=O; b) R=OCH_3, X=H_2

Table 1. Nitration products of 2,3-polymethylene-3,4-dihydroquinazolin-4-ones and quinazolines .

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Reaction product	n	R	Exit %	M.p. ^0C x	R_f^{xx}	Found			Gross formula	Calculated		
						C	H	N		C	H	N
1	2	3	4	5	6	7	8	9	10	11	12	13
V	1	-	73	187-188	0.78	57.1	3.9	18.2	$C_{11}H_9N_3O_3$	57.1	3.9	18.2
VI	2	-	54	179-180	0.64	68.6	4.6	7.0	$C_{12}H_{11}N_3O_3$	58.8	4.5	17.1
VII	3	-	57	104-105	0.64	60.3	5.2	16.3	C13H13N3O3	60.2	5.0	16.2
VIII	4	-	74	182-183	0.94	61.3	5.2	15.3	C14H15N3O3	61.5	5.5	15.4
XI	1	-	90	248-249	0.33	65.8	5.6	20.6	$C_{11}H_{11}N_3O$	65.7	5.5	20.9
XII	1	-	53	184-186	0.12	70.4	7.1	26.1	$C_{11}H_{11}N_3$	70.6	7.0	22.4
XIV	+	-	80	187-189	0.46	60.7	5.3	19.5	$C_{11}H_{11}N_3O_2$	60.8	5.1	19.3
XV	+	-	88	214-216	0.36	50.4	3.7	21.7	$C_{11}H_{10}N_4O_4$	50.4	3.8	21.4
XVI a	+	CH_3	95	265-266	0.28	64.4	5.3	17.1	C13H13N3O2	64.2	50.4	17.3

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16th century	+	OCH_3	88	113-115	0.51	63.9	6.5	17.3	C13H15N3O2	63.7	6.1	17.1

* Note : Compounds I–IV, VI, VII, IX a-z , k-r, XV were recrystallized from acetone; IX a-r from alcohol; XII, XIII from hexane ; XIV from acetone– hexane .

For compounds I, II, V– VIIIa , IXf , c-d, g, j, n-p, x, XV the Rf values were determined in the solvent system chloroform–methanol 1:1, 10:1 (silufol); for XIII chloroform–ether, 15:1 (silufol); IV–X chloroform (aluminum oxide); for III, VIII e, g – chloroform–ether, 14:1 (aluminum oxide); for IX p, XII – chloroform–ether, 9:1 (aluminum oxide).

MATERIALS AND METHODS

Nitration of 2,3-polymethylene-3,4-dihydroquinazolin-4-ones and quinazolines was carried out according to a modified procedure [6].

6-nitro-2,3-trimethylene-3,4-dihydroquinazolin-4-one(V). 5 g (26 mmol) of 2,3-trimethylene-3,4-dihydroquinazolin-4-one are dissolved in 10 ml of sulfuric acid (d=1.84) with stirring and cooling to 0 ° C. A nitrating mixture consisting of 2.5 ml of nitric (d=1.5) and 3.5 ml of sulfuric acid (d=1.84) is added to the reaction mixture in portions with vigorous stirring at such a rate that the temperature of the reaction mixture is below 2 ° C. The mixture is stirred for 1 hour at 5-10 °C , for 1 hour at 20 °C , poured into ice, the formed precipitate is filtered off and thoroughly washed with water. Recrystallization from methanol gives 4.5 g of 6-nitro-2,3-trimethylene-3,4-dihydroquinazolin-4-ones(V) with a mp of 187–188. A mixed melting point with V, obtained by condensation of 5-nitroanthranilic acid with γ- butyrolactam , does not give depression.

Similarly to the above, nitration of 2,3-tetra-, -penta-, hexamethylene-3,4-dihydroquinazaline-4-ones was carried out.

6-nitro-2,3-trimethylene-3,4-dihydroquinazolin-4-one (XIV). 0.33 g (2 mmol) of 2,3-trimethylene-3,4-dihydroquinazoline is dissolved with stirring and cooling to -2-5 ^{0} C in 3 ml of concentrated sulfuric acid. A nitrating mixture consisting of 0.09 ml (2.14 mmol) of nitric acid (d = 1.5) and 0.2 ml of sulfuric acid (d = 1.84) is added to the reaction mixture in portions with vigorous stirring at such a rate that the temperature of the reaction mixture is below 0 ^{0} C. Stir at this temperature for 30 minutes, at 5-10 ^{0} C 1 hour and at 20 ^{0} C for 1 hour, pour into ice, alkalize with ammonia to pH = 8.9. The formed precipitate is filtered off, washed with water, dried. Yield XIV 0.33 g, T (melting point) 187 – 189 (from methanol). IR spectrum: 1620 ($\upsilon_{C=N}$) cm $^{-1}$, 1508 (υ_{NO2}) cm $^{-1}$, molecular weight 217 (mass spectrometry).

6,8-dinitro-2,3-trimethylene-3,4-dihydroquinazoline(XV). From 0.5 g (3 mmol) of 2,3-trimethylene-3,4-dihydroquinazoline and a nitrating mixture consisting of 0.25 ml (6 mmol) of nitric acid (d=1.5) and 0.36 ml of sulfuric acid (d=1.84) 0.56 g of XV are obtained. T(melting point) 214 – 216 (from methanol). IR spectrum: 1625 ($\upsilon_{C=N}$) cm $^{-1}$, 1520 (υ_{NO2}) cm $^{-1}$, molecular weight 262 (mass spectrometry).

6-amino-2,3-trimethylene-3,4-dihydroquinazolin-4-ones[XI]. A mixture of 1.2 g tin chloride in 35 ml concentrated sulfuric acid is added to a solution of 2 g (8.6 mmol) V in

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200 ml alcohol with heating to 80-90 ^{0}C and stirring. The reaction mixture is boiled for 3 hours, cooled, and alkalized with 10% sodium hydroxide solution until a strongly alkaline medium is obtained. Extracted with chloroform and dried with magnesium sulfate. The residue after distilling off the solvent is recrystallized from ethyl acetate. Yield of XI 1.57 g, T(melting point) 248 – 249. IR spectrum: 1625 ($\upsilon_{C=N}$) cm- ^{1} , 1665 ($\upsilon_{C=O}$) cm $^{-1}$, 3339 (υ_{NH2}) cm $^{-1}$ molecular weight 201 (mass spectrometry).

Synthesis of 6-amino-2,3-trimethylene-3,4-dihydroquinazoline[XII]. A mixture of 1 g (5 mmol) of 6-nitro-2,3-trimethylene-3,4-dihydroquinazolin-4-one, 3 g of zinc dust in 30 ml of hydrochloric acid (1:1) was stirred for 8 hours at room temperature. Excess zinc was filtered off, washed with hot water, the filtrate was alkalized with ammonia and extracted with chloroform. The residue after drying (sodium sulfate) and distillation of the solvent was recrystallized from hexane . 0.49 g was obtained, T(melting point) 184 – 186 ^{0}C.

The restoration of IX was carried out in a similar manner to that described above.

6-acetamino-2,3-trimethylene-3,4-dihydroquinazolin-4-one[XVI]. 1 g (5 mmol) of 6-amino-2,3-trimethylene-3,4-dihydroquinazolin-4-one is dissolved in 10 ml of acetic anhydride and heated for 1 hour at a temperature of 50–60 ^{0}C. The precipitate that forms upon cooling is filtered off, thoroughly washed with water and dried. Yield 1.15 g, T(melting point) 265–266 ^{0}C (from alcohol).

6-Carbomethoxyamino-2,3-trimethylene-3,4-dihydroquinazoline hydrochloride [XVI]. To a solution of 0.45 g (2.4 mmol) of 6-amino-2,3-trimethylene-3,4-dihydroquinazoline in 8 ml of chloroform is added 0.4 ml (4.7 mmol) of methyl chlorocarbonate. After 30 minutes, the formed precipitate is filtered off, washed with ether, and dried. 0.6 g of the acid chloride is obtained . T (melting point) 290 ^{0}C (decomp .).

Results and discussion

The synthesis of the target compounds is shown in Scheme 1. The starting material 2,3-trimethylene-3,4-dihydroquinazoline (3) was prepared according to the described procedure [21] by refluxing N- (trimethoxybenzoyl)-3-methoxyanthranilic acid with acetic anhydride followed by reaction with hydrazine to give the compound 33-amino-8-methoxy-2-trimethoxyphenylquinazoline-4. The IR spectra of the compound showed absorption bands at 3221.35, 3387.83, and 1655.19 cm due to the stretching vibrations of the −1 NH 2 and CO groups , respectively, and 1 H NMR resulted in the appearance of a singlet consisting of two protons for the NH 2 group at 5.842.

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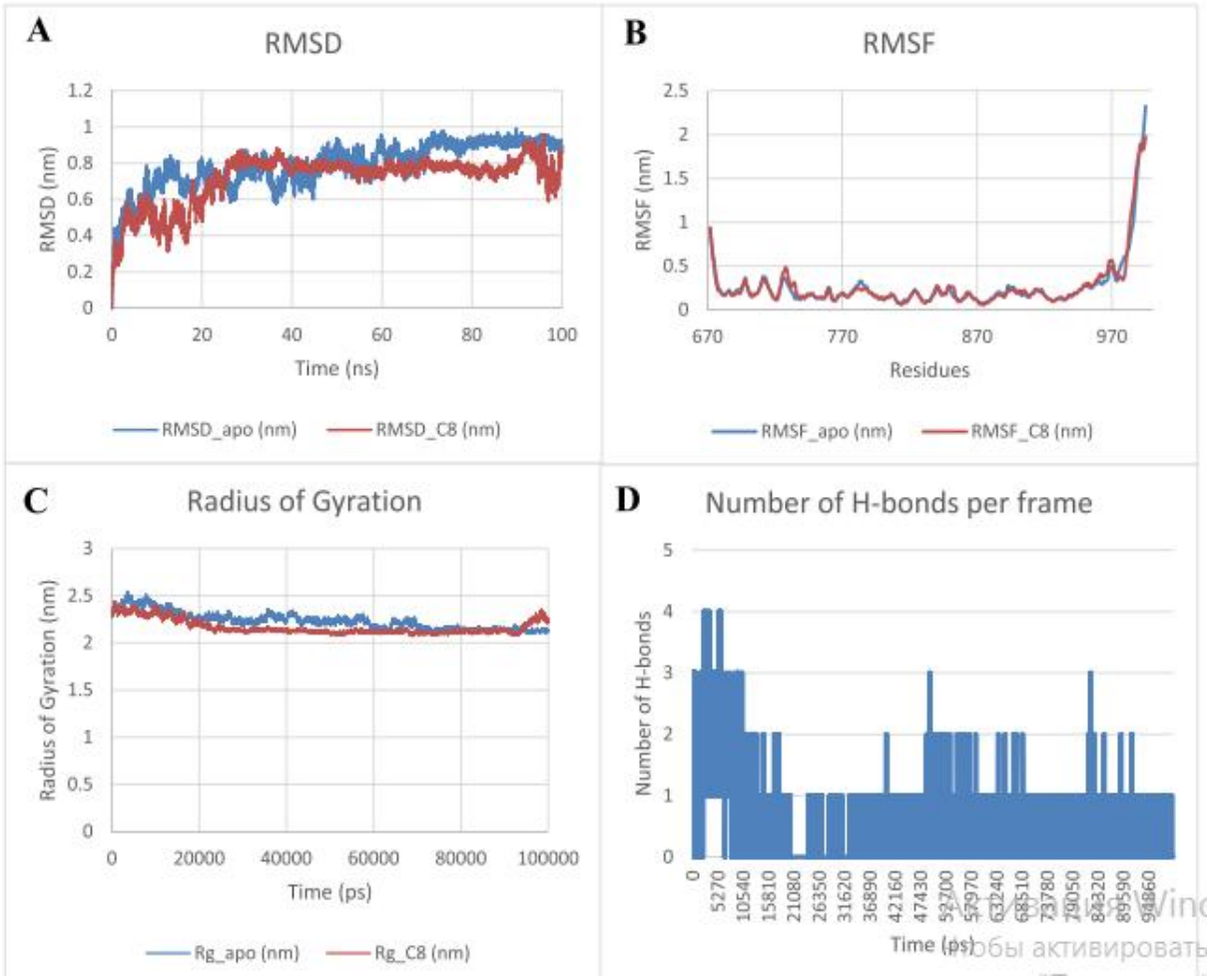

Compound 3 reacts with formamide to form 8-methoxy-2-trimethoxyphenylquinazolin-4(3 H)-one (5), the latter compound shows a broadened band for CO at 1655.67 cm−1 and 1 H NMR shows a singlet peak for NH group at 8.292 ppm . The compound reacts with ammonia to form benzamide derivative 6, the IR spectrum of compound 6 shows broadened vibrational bands for NH and NH at 3193.13, 3367.91 and 3389.62 and broadened bands at 1672.66 and 1663.23 for two CO groups. In addition, 1 H NMR data showed a singlet peak for NH group at 6.069 ppm . and a singlet peak for the NH fragment at 9.782 ppm . The compound reacts with hydrazine hydrate in the presence of ethanol to form a hydrazide derivative . The compound showed characteristic CO bands diamide at 1670.63 and 1655.19 and NH , NH . Absorption at 3376.45, 3382.21, 3175.43 and 3295.34 cm−1, in addition to a singlet H NMR peak at 4.479 ppm for NH and two singlets for NH diamide at 10.134 and 10.167 ppm . Compound 7 reacts with benzenesulfonyl chloride to form a compound 83366.45, 3372.52 and 3385.43 cm−1 with absorption bands of NH groups and a CO band at 1670.65 and 1655.37 cm−1 in the IR spectrum, as well as singlets at 8.5497 and 12.615 ppm for NH protons .1 H NMR. In addition, the compound reacts with 4-benzenesulfonyl chloride to form a compound. Singlet 1 H NMR for NH at 11.885 ppm . due to CO and NH , respectively, around −1 with absorption lines at 1655.82 and 3385.56 cm. Furthermore, the compound reacts with methylamine to form 3-methyl-8-methoxy-2-trimethoxyphenylquinazolin-4(3 H)-one (10), the latter compound being confirmed by the presence of the CH group at 317.08 and 2.637 ppm by 13 C NMR and 1 H

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NMR, respectively. Compound 33 reacts with 4,5-trimethoxyaniline to form 3-(3,4,5-trimethoxyphenyl)-8-methoxy-2-trimethoxyphenylquanazoline-4, which is confirmed by the presence of separate peaks at 56.18, 59.56 and 60.23 ppm . and 3.624, 3.768 and 3.964 ppm , due to one methoxy group and two trimethoxy groups.

6-nitro-2,3-trimethylene-3,4-dihydroquinazolin-4-one (8) A mixture of equimolar amounts of 3-aminoquinazoline derivative (4) and isatin (10 mmol) in glacial acetic acid (5 ml) was heated under reflux for 2 h, then poured into ice water. The resulting solid was filtered, washed with water and recrystallized from ethanol to give methyl orange crystals; yield 75%; mp 220–222°C; IR (cm−1): 3267 (NH), 3098-3067 (CH aromatic), 2920 (CH aromatic), 1720 (C = O), 1686 (C = O), 1612 (NH bending), 1516-1489 (C = C); 1 H NMR (DMSO-d 6, 400 MHz, d , ppm): 4.91 (s, 2 H , CH 2 O), 6.73 (d , J = 8.40 Hz, 2 H , aromatic H), 6.96 (d , J = 8.00 Hz, 2 H , aromatic H), 7.11 (t , J = 7.56 Hz, 1 H , aromatic H), 7.35 (d , J = 8.80 Hz, 1 H , aromatic H), 7.40 (d , J = 7.68 Hz, 1 H , aromatic H), 7.51 (d , J = 8.80 Hz, 1 H , aromatic H), 7.58 (d , J = 8.04 Hz, 1 H , aromatic H), 8.25 (d , J = 7.80 Hz, 1 H , aromatic H), 8.45 (s, 1 H , aromatic H), 11.31 (s, 1 H , NH , exchanged with D 2 O); Analysis calculated. For C 23 H 14 BrIN 4 O 3; C, 45.09; H, 2.32; Cb , 9.30; Found; C, 46.12; H, 2.43; H.

4-nitro-2,3-trimethylene-3,4-dihydroquinazolin-4-one white crystals; yield 87%; mp 284–286°C; IR (cm−1): 3336 (NH), 3094-3039 (CH aromatic), 2920 (CH aliphatic), 1659 (2 C = O), 1597-1489 (C = C); 1 H NMR (DMSO- d 6, 100 MHz, d , ppm): 1.35-1.49 (m, 6 H , piperidine 3 CH 2), 2.45 (br s , 4 H , piperidine N (CH 2) 2), 2.87 (s, 2 H , CH 2 N), 3.17 (s, 1 H , NH replaced by D 2 O), 4.93 (d, J = 15.64 Hz, 1 H , CH 2 O), 5.20 (d, J = 15.60 Hz, 1 H , CH 2 O), 6.93 (d, J = 8.92 Hz, 2 H , aromatic H), 7.29 (d, J = 8.60 Hz, 1 H , aromatic H), 7.40 (d, J = 8.88 Hz, 2 H , aromatic H), 7.91 (d , J = 8.60 Hz, 1 H , aromatic H), 8.33 (s, 1 H , aromatic H); 13 C NMR (DMSO- δ 6, 100 MHz, d , ppm) : 24.5 (piperidine CH2), 26.0 (piperidine 2CH2), 54.8 (piperidine N (CH2)2), 63.8 (CH2N), 66.3 (CH2O) , 90.3 (CI) , 112.5, 117.50, 124.1, 129.5 132.4, 134.7, 141.4, 146.4, 155.2, 158.1 (aromatic carbons), 158.3 (C = O), 172.9 (C = O); The analysis is calculated for C 22 H 22 BrIN 4 O 3; C, 44.22; N, 3.68; N, 9.38; Found: C, 44.47; N, 3.84; N, 9.47.

To a stirred solution of 3-aminoquinazoline derivative (4) (2.37 g, 5 mmol) in dry DMF (5 ml) was added dropwise chloroacetyl chloride (0.65 ml, 5.5 mmol) and stirring was continued at room temperature overnight. The solution was poured onto crushed ice with vigorous stirring, then filtered. The resulting precipitate was washed with water and recrystallized from ethanolchloroform to give yellow crystals; yield 81%; mp 198–200°C; IR spectrum (cm−1): 3387 (NH), 3194 (CH aromatic), 2990 (CH aliphatic), 1725 (C = O), 1685 (C = O), 1609 (NH bend), 1489 (C = C); 1 H NMR (DMSO- d 6, 400 MHz, d , ppm): 4.40 (s, 2 H , CH 2 O), 5.04 (d , J = 8.40 Hz, 1 H , CH 2 Cl), 5.11 (d , J = 8.40 Hz, 1 H , CH 2 Cl), 6.98 (d , J = 8.92 Hz, 2 H , aromatic H); 7.45-7.50 (m, 3 H , aromatic H); 8.18 (d , J = 8.60 Hz, 1 H , aromatic H), 8.43 (s , 1 H , aromatic H).

Conclusions

1. For the first time, the nitration reaction of 2,3-polymethylene-3,4-dihydroquinazolin-4-ones and quinazolines was studied .
2. It turned out that this results in the formation of reaction products of electrophilic substitution of the aromatic ring.
3. The possibility of the reaction occurring with the substitution of the hydrogen atom of the aromatic ring in position 6 is shown.

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4. It was found that it was not possible to introduce a second nitro group into the quinazoline ring earlier when using an excess of the nitrating mixture (1:4) and under relatively harsh conditions (heating to 90-100%).

5. Nitration of 2,3-polymethylene-3,4-dihydroquinazolines shows that when using a 1:1 reagent ratio, a mono nitro product, 6-nitro derivative, is formed, and at 1:2, a dinitro product, 6,8-dinitro-2,3-trimethylene-3,4-dihydroquinazoline, is formed.

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