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Note

Reaction of amines with nitrones derived from chromone-3-carbaldehyde

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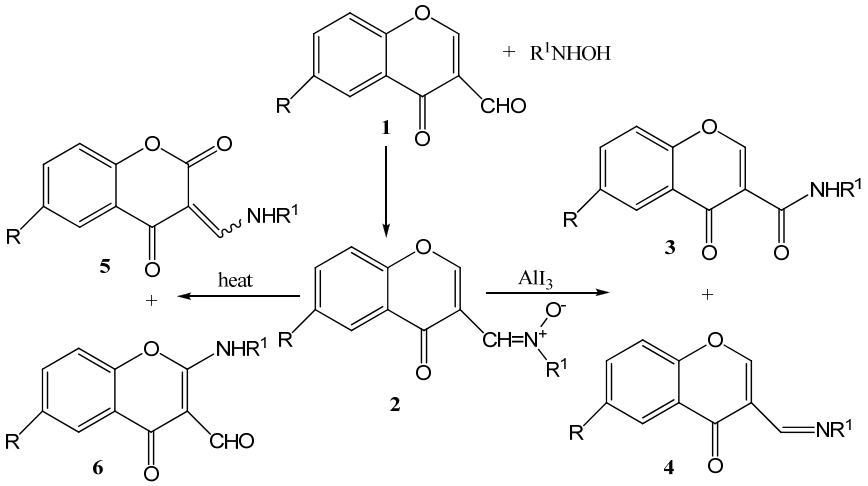
Nitrone **2** derived from 3-formylchromone **1** produces 3-aminomethylene-2-iminochroman-4-one **8** on reaction with primary amine **7**, chromenoquinoline **14** with piperidine and dihydrotetraaza[14]annulene **15** with *o*-phenylenediamine.

Keywords: Nitrone, 3-formylchromone, 2-iminochroman-4-one, chromenoquinoline, 1-benzopyran

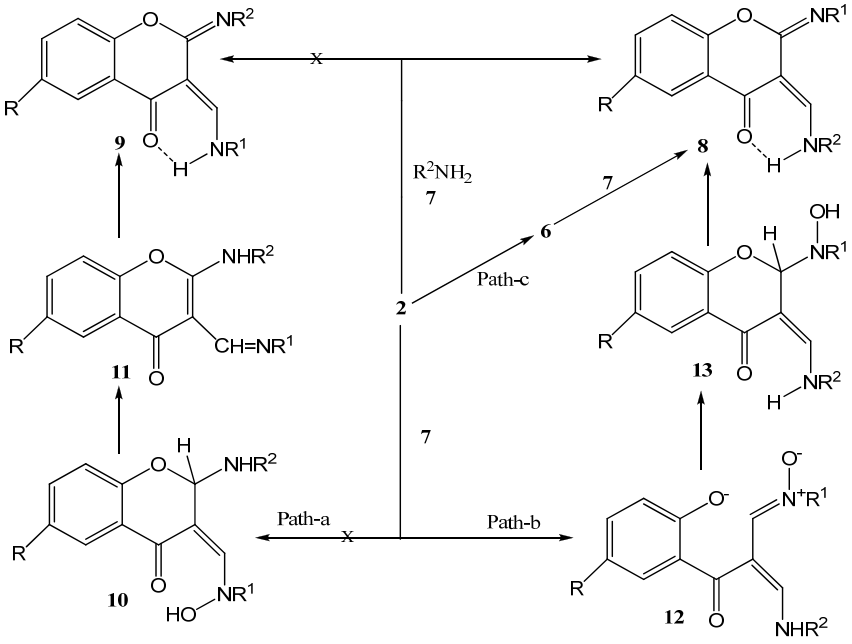
During the last decade the nitrones (C-[3-chromonyl]-*N*-alkyl/arylnitrones) **2** derived from chromone-3-carbaldehyde **1** have been widely studied and many interesting results have come out from the combined effect of nitrone and chromone moieties. Since the preparation of nitrone **2** (Ref. 1), its 1,3-dipolar nature was exploited for the synthesis of isoxazolidines linked to chromone ring (Ref. 2, 3). Later microwave-assisted cycloaddition reaction (Ref. 4), regio- and stereoselectivities of those reactions (Ref. 5) and tandem reorganization of 1,3-dipolar adduct of allenic esters (Ref. 6) were also studied. AlI_3-induced rearrangement of **2** to chromone-3-carboxamide **3** and deoxygenation of **2** to imine **4** (Ref. 7), thermal rearrangements of **2** to chroman-2,4-dione **5** and 2-amino-3-formylchromone **6** (Ref. 8) were also reported (**Scheme I**). In the last few years an easy route for the synthesis of **2** (R^1 = alkyl or aryl) from aldehyde **1** and R^1NO_2 (Ref. 9, 10), a one-pot synthesis of aminoaldehyde **6** (R^1 = alkyl or aryl) from aldehyde **1** (Ref. 9) and differences in the reactivities of *N*-alkyl and *N*-aryl nitrones **2** (Ref. 10) have been developed. The thermal rearrangement of nitrones **2** to **5** and **6** was proved to be a solvent-dependent process and selectivity over the formation of the desired product was achieved (Ref. 11). Although 1,3-dipolar addition and rearrangement reactions of **2** have been studied in detail, no attention have been given for the reactivities of **2** towards nucleophiles. Reactions of aldehyde **1** with nucleophiles (Ref. 12, 13) usually proceed by opening of the pyran ring or by 1,2-addition to aldehyde function, whereas, 3-aryliminomethylchromone **4** facilitates nucleophilic addition rather than pyran ring fission (Ref. 14). So it was of interest to study how the nitrone **2** would behave towards nucleophiles. The results of the reactions of different amines with **2** are reported herein.

Results and Discussion

A mixture of nitrones **2** and primary amines **7** was heated under reflux in ethanol for 2-4 hr. On concentration, the reaction-mixture produced fine crystalline yellow solids **8** in very good yields (80-90%, **Table I**). Initially the reactions were carried out using *N*-arylnitrones **2** with arylamines **7** (**Table I**, entries 1-3). From the ^{1}H NMR spectra of their products it was observed that a very low field signal at δ13.9-14.0 and another signal at δ 9.0-9.1 are the most important information for the assignment of the structures. Originally the structures were assigned to be **9** based on the spectral data and also from the consideration of 1,4-addition of nucleophiles to the nitrones **2**, as was observed for the Schiff bases **4** (Ref. 14). Formation of **9** was considered by the addition of **7** to **2** (→ **10**) followed by the dehydration (→ **11**) and 1,5-H shift (**Scheme II**, Path-a). Looking at the basic structure of **9**, it appeared to form from the reaction of 2-(*N*-substitutedamino-3-formylchromone) **6** with appropriate amino compounds **7**. The reaction of **6** ($R^1 = C_6H_4Me$-*p*) with *p*-toluidine indeed produced the compound **9** ($R^1 = R^2 = C_6H_4Me$-*p*) in excellent yield. But this correlation does not hold well when the alkyl or aryl substituent on nitrogen of nitrone **2** differs from the alkyl or aryl parts of amine **7**. Reaction of nitrone **2** (R = H, R^1 = Ph) with ethyl amine (**Table I**, entry 4) yielded a compound, which showed a peak in its ^{1}H NMR spectrum at δ 11.79 for the H-bonded NH instead of at around δ 14.00 as was observed for the products obtained by the reaction of **2** with aromatic amines (entries 1-3). This relative upfield shift of H-bonded NH has some analogy with an earlier report (Ref. 9), where the ^{1}H NMR spectra of **6** revealed that N-H protons for *N*-aryl derivatives **6** (R^1 = aryl) appear at δ 12.4-12.5, whereas those of



Scheme I



For **2-13**

a: R = H, R^1 = Ph, R^2 = $C_6H_4Me(p)$
b: R = Me, R^1 = R^2 = $C_6H_4Me(p)$
c: R = Me, R^1 = $C_6H_4Me(p)$, R^2 = Ph
d: R = H, R^1 = Ph, R^2 = Et
e: R = Me, R^1 = $C_6H_4Me(p)$, R^2 = Et
f: R = Me, R^1 = Et, R^2 = $C_6H_4Me(p)$
g: R = Me, R^1 = R^2 = Et

Scheme II

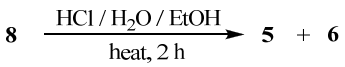
N-alkyl derivatives **6** (R^1 = alkyl) appear at δ 10.5-10.6. Considering these results, it was thought that the product might be **8** (R^1 = Ph, R^2 = Et) instead of **9** (R^1 = Ph, R^2 = Et). Indeed this assumption was proved to be correct when compound **8d** was obtained by heating **6a** (R = H, R^1 = Ph) with $EtNH_2$. A few more cases were considered to corroborate the above results (*vide* experimental). The only exceptional case was observed in the ^{1}H NMR spectrum of **8f**, where H-bonded NH appears at δ 12.05. If rearrangement is not considered in this case, the product should be identical with **8e**, which is not the fact. Again compound **8f** was found to be identical with the product of the reaction of **6f** (R = Me, R^1 = Et) with *p*-toluidine. It should be mentioned here that primary aromatic amines react with aldehyde **1** or Schiff base

4 to produce 2-arylamino-3-arylaminomethylene-2*H*-chromen-4-one (Ref. 14), a reduced form of **8**.

The formation of **8** may be rationalised in two ways: (i) nucleophilic addition of **7** at the 2-position of chromone ring followed by the pyran ring opening to form **12**, which on ring closure (→ **13**) and subsequent dehydration produces **8** (**Scheme II**, path-b); or (ii) *via* the rearranged product **6**, which reacts with **7** to produce **8** (**Scheme II**, path-c). The formation of **8** *via* **6** (path-c) has been supported by carrying out the conversion of **6** to **8** under identical reaction condition, but we could not provide any evidence supporting path-b. On acidic hydrolysis, compound **8** produces a mixture of 3-(*N*-substitutedamino-methylene)chroman-2,4-dione **5** (Ref. 15) and 2-(*N*-substitutedamino)-3-formylchromone **6** (**Scheme III**).

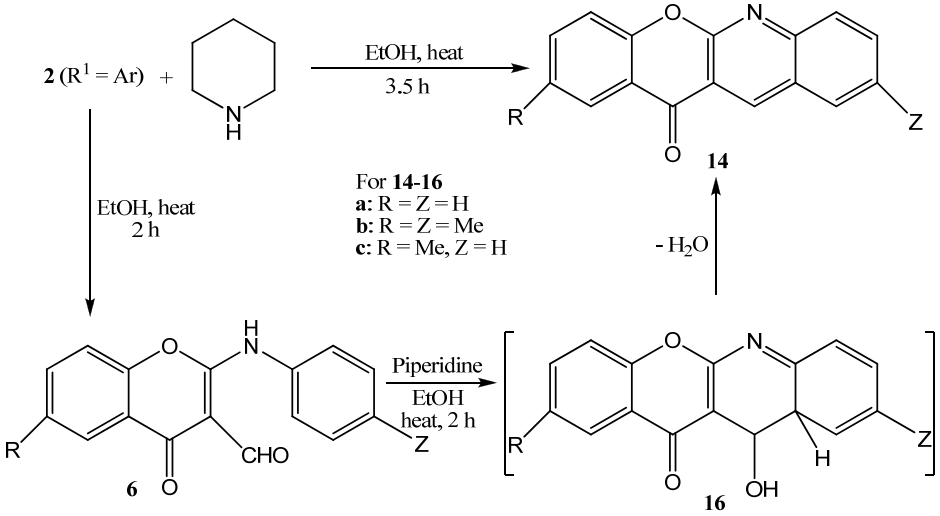
Table I — Products **8** obtained by the reaction of primary amines **7** with nitones **2**

Entry	R	R^1	R^2	Product	Yield (%)	m.p (°C)
1	H	Ph	C_6H_4Me-*p*	8a	85	196-98
2	Me	C_6H_4Me-*p*	C_6H_4Me-*p*	8b	84	214
3	Me	C_6H_4Me-*p*	Ph	8c	82	192-94
4	H	Ph	Et	8d	79	158-60
5	Me	C_6H_4Me-*p*	Et	8e	72	160-62
6	Me	Et	C_6H_4Me-*p*	8f	63	146-48
7	Me	Et	Et	8g	97	146

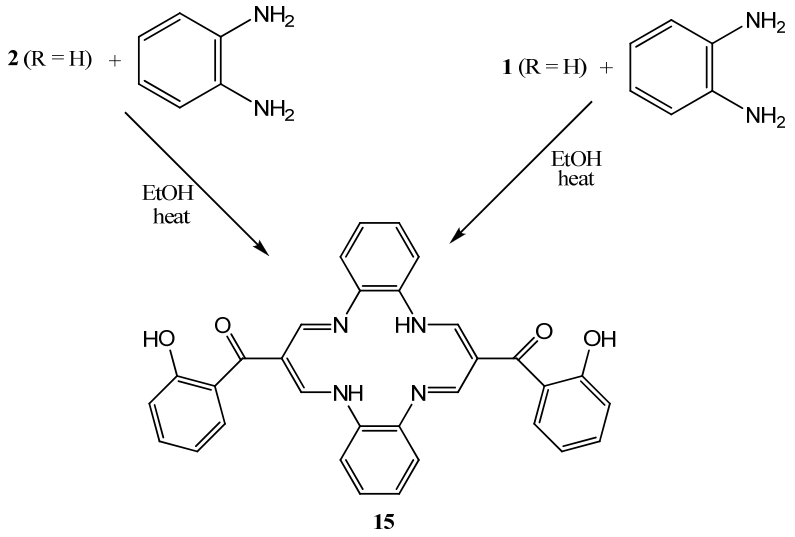


Scheme III

On heating an ethanolic solution of nitrone **2** with *N*-methylaniline for 3 hr, the rearranged product **6** was obtained. Compound **6** was reported to form from **2** by heating in ethanol alone (Ref. 11). So, *N*-methylaniline causes no effect on nitrone **2**. Schiff base **4** was also reported to remain unchanged when treated with *N*-methylaniline (Ref. 14). Although *N*-methylaniline does not cause any change on **2**, piperidine acts differently. On heating nitrone **2** with equivalent amount of piperidine in ethanol chromeno-quinolines **14** (Ref. 9) was produced (**Scheme IV**). Earlier this compound **14** was prepared by treating **6** (R^1 = aryl) with H_2SO_4 (Ref. 5, 9). Piperidine-induced transformation of **2** to **14** was also considered *via* the rearranged product **6**. To justify it, compound **2** was heated under reflux in ethanol for 2 hr till the complete conversion of **2** to **6** (observed by TLC). Under this condition piperidine was added to this reaction-mixture and was heated under reflux for another 2 hr to get **14** (**Scheme IV**). Compound **6** (R^1 = aryl) was also heated with piperidine in ethanol for 2 hr and indeed compound **14** was obtained in good yield. So, a one-pot synthesis of chromenoquinoline **14** from **2** has been achieved by an easy method compared to the previously reported two-step process (Ref. 5, 9). To make some generalization for the base-induced transformation of **6** to **14**, compound **6** (R^1 = aryl) was treated with pyridine in ethanol for 7.5 h, but compound **6** remains unchanged. Surprisingly, on heating **2b** [R = Me, R^1 = C_6H_4Me(*p*)] with Et_3N in ethanol for 7 hr and subsequent work-up afforded a yellow compound **8b** in very low yield. Formation of **8** may be considered in this way. Hydrolysis of nitrone **2** produces *N*-substituted hydroxylamine,



Scheme IV



Scheme V

which generates some amine **7** by disproportionation. This amine subsequently reacts with **2** to produce **8**.

So, the conversion of **2** to **14** may be rationalized by considering the thermal rearrangement of **2** to **6**, (Ref. 8, 11) followed by its base-induced cyclisation to **14** *via* **16** (**Scheme IV**). Higher basicity of piperidine compared to pyridine or triethylamine may be responsible for this differential behaviour.

Reactions of *o*-phenylenediamine with nitrones **2** were also studied. It was found to produce dihydrotetraaza[14]annulene **15**, as was reported to obtain from the reaction of aldehyde **1** and *o*-phenylenediamine (Ref. 16) (**Scheme V**).

Conclusion

Primary amines react with nitrones **2** to produce 2-alkyl/arylimino-3-alkyl/arylaminomethylenechroman-4-one **8**, piperidine transforms **2** into chromenoquinoline **14** in a one-pot reaction and *o*-phenylenediamine reacts with **2** to produce **15,** as was obtained from the reaction of aldehyde **1** and *o*-phenylenediamine (Ref. 16). So, nitrones **2** have a little similarity with aldehyde **1** or Schiff base **4** in their reactivites towards different amines.

Experimental Section

General. Melting points are uncorrected. ^{1}H NMR spectra were recorded on a Bruker 300 MHz spectrometer in $CDCl_3$, IR spectra in KBr on a Beckmann IR 20 A instrument and elemental analysis on a Perkin Elmer 240C elemental analyser. Light petroleum refers to the fraction with distilling range 60-80^{o}C unless stated otherwise.

Reaction of nitrone 2 with primary amine 7 for the synthesis of 8: **General procedure**. A mixture of nitrone **2** (0.5 mmole) and amine **7** (0.5 mmole) was heated under reflux in ethanol (25 mL) for 2 hr. The reaction-mixture on concentration and cooling produced a yellow solid, which on further crystallization from alcohol afforded yellow solids **8**.

2-(*N*-phenyl)imino-3-(*p*-tolylaminomethylene)-chroman-4-one 8a: Yellow fine crystalline compound (150 mg, 85%), m.p. 196-98^{o}C; IR: 3450, 2800, 1675, 1600 cm^{-1}; ^{1}H NMR: δ 13.96 (1 H, d, exchangeable, *J* = 9.9 Hz, NH), 9.08 (1 H, d, *J* = 9.9 Hz, vinylic H), 8.18 (1 H, brd, *J* = 7.5 Hz, 5-H), 7.54 (1 H, brt, *J* = 7.3 Hz, 7-H), 7.45-7.40 (2 H, m, ArH), 7.37-7.34 (2 H, m, ArH), 7.30-7.13 (7 H, m, ArH), 2.37 (3 H, s, CH_3); Anal. Calcd. for $C_{23}H_{18}N_2O_2$: C, 77.95; H, 5.12; N, 7.90. Found: C, 77.80; H, 5.00; N, 7.78%.

6-methyl-3-*p*-tolylaminomethylene-2-(*N*-*p*-tolyl)-iminochroman-4-one 8b: Yellow fine crystalline fluffy compound (160 mg, 84%), m.p. 214^{o}C; IR: 3448, 2850, 1664,1624, 1610 cm^{-1}; ^{1}H NMR: δ 14.04 (1 H, brs, exchangeable, NH), 9.08 (1 H, brs, vinylic H), 7.97 (1 H, brs, 5-H), 7.34 (1 H, brd, *J* = 8.3 Hz, 7-H), 7.30-7.21 (8 H, m, ArH), 7.07 (1 H, d, *J* = 8.3 Hz, 8-H), 2.41 (3 H, s, CH_3), 2.39 (3 H, s, CH_3), 2.37 (3 H, s, CH_3); Anal. Calcd. for $C_{25}H_{22}N_2O_2$: C, 78.51; H, 5.80; N, 7.32. Found: C, 78.35; H, 5.75; N, 7.20%.

6-methyl-3-(phenylaminomethylene)-2-(*N*-*p*-tolyl)-iminochroman-4-one 8c: Yellow fine crystalline fluffy solid (150 mg, 82%), m.p. 192-94^{o}C; IR: 3436, 2921, 1660, 1616, 1569 cm^{-1}; ^{1}H NMR: δ 14.04 (1 H, d, *J* = 7.4 Hz, exchangeable, NH), 9.11 (1 H, d,

J = 7.4 Hz, vinylic H), 7.97 (1 H, d, J = 1.9 Hz, 5-H), 7. 44-7.39 (2 H, m, ArH), 7.35 (1 H, dd, J = 8.4, 1.9 Hz, 7-H), 7.31 (2 H, d, J = 8.4 Hz, ArH), 7.29 (2 H d, J = 8.4 Hz, ArH), 7.26-7.21 (3 H, m, ArH), 7.08 (1 H, d, J = 8.4 Hz, 8-H), 2.41 (3 H, s, CH_3), 2.39 (3 H, s, CH_3); Anal. Calcd. for $C_{24}H_{20}N_2O_2$: C, 78.24; H, 5.47; N, 7.60. Found: C, 78.30; H, 5.41; N, 7.45%.

3-Ethylaminomethylene-2-(*N*-phenyl)iminochroman-4-one 8d: Yellow crystalline compound (115 mg, 79%), m.p. 158-60°C; IR: 3435, 2931, 1664, 1616, 1571 cm^{-1}; ^1H NMR: δ 11.79 (1 H, brs, exchangeable, NH), 8.59 (1 H, brs, vinylic H), 8.11 (1 H, dd, J = 7.8, 1.5 Hz, 5-H), 7.48 (1 H, dt, J = 8.4, 1.5 Hz, 7-H), 7.39-7.34 (2 H, m, ArH), 7.23-7.10 (4 H, m, ArH), 7.02 (1 H, d, J = 8.4 Hz, 8-H), 3.60 (2 H, q, J = 7.2 Hz, CH_2Me), 1.39 (3 H, t, J = 7.2 Hz, CH_3); Anal. Calcd. for $C_{18}H_{16}N_2O_2$: C, 73.96; H, 5.52; N, 9.58. Found: C, 74.10; H, 5.61; N, 9.50%.

3-Ethylaminomethylene-6-methyl-2-(*N*-*p*-tolyl)-iminochroman-4-one 8e: Yellow crystalline compound (116 mg, 72%), m.p. 160-62°C; IR: 3440, 2900, 1650,1620 cm^{-1}; ^1H NMR: δ 11.89 (1 H, brs, exchangeable, NH), 8.57 (1 H, d, J = 12.6 Hz, vinylic H), 7.89 (1 H, brs, 5-H), 7.28 (1 H, brd, J = 8.1 Hz, 7-H), 7.17 (2 H, d, J = 8.1 Hz, ArH), 7.11 (2 H, d, J = 8.1 Hz, ArH), 6.90 (1 H, d, J = 8.1 Hz, 8-H), 3.60 (2 H, q, J = 7.2 Hz, CH_2Me), 2.37 (6 H, s, 2×$ArCH_3$), 1.38 (3 H, t, J = 7.2 Hz, CH_3); Anal. Calcd. for $C_{20}H_{20}N_2O_2$: C, 74.98; H, 6.29; N, 8.74. Found: C, 75.11; H, 6.40; N, 8.65%.

2-(*N*-Ethyl)imino-6-methyl-3-(*p*-tolylaminomethylene)chroman-4-one 8f: Yellow crystalline compound (100 mg, 63%), m.p. 146-48°C; IR: 3448, 2907, 1654,1630 cm^{-1}; ^1H NMR: δ 12.05 (1 H, brs, exchangeable, NH), 9.17 (1 H, brs, vinylic H), 8.02 (1 H, brs, 5-H), 7.36 (1 H, brd, J = 8.1 Hz, 7-H), 7.19 (1 H, d, J = 8.1 Hz, 8-H), 7.16-7.12 (4 H, m, ArH), 3.65 (2 H, q, J = 7.2 Hz, CH_2Me), 2.43 (3 H, s, CH_3), 2.36 (3 H, s, CH_3), 1.40 (3 H, t, J = 7.2 Hz, CH_3); Anal. Calcd. for $C_{20}H_{20}N_2O_2$: C, 74.98; H, 6.29; N, 8.74. Found: C, 74.85; H, 6.18; N, 8.88%.

3-Ethylaminomethylene-2-(*N*-ethyl)imino-6-methylchroman-4-one 8g: White crystalline fluffy solid (125 mg, 97%), m.p. 146°C; IR: 3448, 2930, 1627, 1618 cm^{-1}; ^1H NMR: δ 11.99 (1 H, brs, exchangeable, NH), 8.75 (1 H, brs, vinylic H), 7.96 (1 H, d, J = 1.8 Hz, 5-H), 7.32 (1 H, dd, J = 8.4, 1.8 Hz, 7-H), 7.12 (1 H, d, J = 8.4 Hz, 8-H), 3.52-3.62 (4 H, m, 2×CH_2Me), 2.41 (3 H, s, $ArCH_3$), 1.32 (3 H, t, J = 7.1 Hz, CH_3), 1.28 (3 H, t, J = 6.9 Hz, CH_3); Anal. Calcd. for $C_{15}H_{18}N_2O_2$: C, 69.74; H, 7.02; N, 10.84. Found: C, 69.61; H, 6.91; N, 10.65%.

Treatment of 6 with primary amines 7. On heating an ethanolic solution (25 mL) of **6a** (R=H, R^1=Ph) (0.5 mmole) with amine **7a** [$R^2 = C_6H_4Me(p)$] (0.5 mmole) for 2 hr under reflux and on subsequent concentration and cooling, the reaction-mixture produced a yellow crystalline compound **8a** (160 mg, 77%). Similarly, compound **8b** (180 mg, 94%), **8d** (130 mg, 89%), **8e** (150 mg, 94%) and **8f** (140 mg, 87%) were obtained starting from appropriate **6** (0.5 mmole) and **7** (0.5 mmole).

Hydrolysis of 8: Compound **8** (0.5 mmole) was heated under reflux in ethanol (20 mL) containing concentrated HCl (4 drops) and H_2O (4 drops) for 2 hr. After usual work-up and chromatographic separation using benzene as eluent, compounds **5b** (40 mg, 30%, Ref. 15) **6b** (30 mg, 20%, Ref. 9); **5** (R^1 = Et) (35 mg, 24%, Ref. 11), **6d** (25 mg, 19%, Ref. 9) were isolated from the reaction-mixture of **8b** and **8d**, respectively. Compound **5b, 5** (R^1 = Et) and **6b,d** are identical in all respects with the authentic samples (Ref. 9, 11, 15).

Treatment of nitrone 2 with piperidine: **General procedure**. An ethanolic solution (25 mL) of nitrone **2** (0.5 mmole) and piperidine (40 mg, 0.5 mmole) was heated under reflux for 3.5 hr. On concentration and cooling, the reaction-mixture produced yellow solid. It was filtered off and recrystallised from benzene-petroleum ether to afford yellow crystalline solids **14a** (100 mg, 81%), **14b** (115 mg, 84%) and **14c** (100 mg, 76%). Compounds **14a-c** are identical in all respects with those produced *via* the two-step procedure (Ref. 9).

Conversion of 6 (R^1=aryl) to 12-oxo-12*H*-chromeno[2,3-b]quinolines 14. A mixture of appropriate **6** (0.5 mmole) and piperidine (45 mg, 0.5 mmole) in ethanol (20 mL) was heated under reflux for 2 hr. Subsequently, the reaction-mixture was concentrated and cooled to produce a yellow crystalline compound. It was recrystallised from benzene-petroleum ether to afford **14a** (110 mg, 89%), **14b** (125 mg, 91%) and **14c** (115 mg, 88%, Ref. 9).

Reaction of Et_3N with nitrone 2b [R=Me, R^1=$C_6H_4Me(p)$]. Et_3N (50 mg, 0.5 mmole) was added to an ethanolic solution (25 mL) of **2b** (145 mg, 0.5 mmole) and the resultant mixture was heated under reflux for 7 hr. Solvent was removed from the reaction-mixture under reduced pressure. Cold water (10 mL) was added to the concentrate to produce a yellow semisolid mass, which was extracted with

$CHCl_3$ (2×10 mL), washed with water (2×15 mL), dried over Na_2SO_4 and was chromatographed over silica gel using benzene as eluent to produce a yellow crystalline compound **8b** (30 mg, 15%).

Reaction of *o*-phenylenediamine with 2 (R = H, R^1=Ph/*p*-tolyl/ Me). On heating an equimolar mixture of **2** (0.5 mmole) and *o*-phenylenediamine (55 mg, 0.5 mmole) in ethanol (25 mL) for 2 hr, a solid began to separate. It was filtered off and recrystallised from $CHCl_3$-petroleum ether to produce **15** (90 mg, 68%), **15** (100 mg, 76%), and **15** (75 mg, 57%), respectively from the reactions of **2** (R=H, R^1=Ph), **2** [R=H, R^1=$C_6H_4Me(p)$] and **2** (R=H, R^1=Me). Compound **15** is identical with the corresponding authentic sample prepared from the reaction of **1** and *o*-phenylenediamine (Ref. 16).

2,3:9,10-Dibenzo-6,13-disalicyloyl-1,8-dihydro-1,4,8,11-tetraaza[14]annulene 15. Red crystalline compound, m.p. 226-28°C; IR: 3540, 3310, 3120, 1695, 1615 cm^{-1}; ^1H NMR: δ 14.56 (2 H, brs, exchangeable, 2×OH), 11.39 (2 H, brs, exchangeable, 2×NH), 8.60 (2 H, s, 2×vinylic H), 8.58 (2 H, s, 2×vinylic H), 7.58 (2 H, brd, *J* = 7.8 Hz, 2×6′-H), 7.49 (2 H, brt, *J* = 8.0 Hz, 2×4′-H), 7.27-7.24 (4 H, m, ArH), 7.17-7.13 (4 H, m, ArH), 7.09 (2 H, brd, *J*=8.4 Hz, 2×3′-H), 6.95 (2 H, brt, *J*=7.6 Hz, 2×5′-H).

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