An efficient synthesis of quinalone-3-\((N\text{-phenylpyrazoles})\) and quinalone-3-cyclohexadienone derivatives†

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An efficient synthesis of quinalone-3-(\(N\text{-phenylpyrazoles})\) 5 and quinalone-3-(cyclohexadienones) 8 has been described. 2-Chloroquinoline-3-carboxaldehyde 1, on reaction with 4N HCl gives 3-formyl-2(1\(H\))-quinalone 2. The latter is reacted with acetophenone in the presence of NaOH as base to yield the corresponding quinalone-3-chalcone 3 which on treatment with phenylhydrazine gives 3-(dihydropyrazolo)quinolone 4. Dehydrogenation of 4 with chloranil yields the corresponding 5. Compound 3 on bromination yields the dibromo derivative 6 which on treatment with phenylhydrazine directly yields 5. On the other hand, 3 on treatment with acetoacetanilide in methanol using acetic acid as a catalyst affords the corresponding quinalone-3-(cyclohexenone) derivatives 7 which on dehydrogenation with chloranil, affords the corresponding 8 in excellent yield.

Keywords: 2-Chloroquinoline-3-carboxaldehyde, 3-formyl-2(1\(H\))-quinalone, phenylhydrazine, acetoacetanilide, chloranil

The synthesis of quinoline derivatives\(^1\) is of great interest in organic chemistry. The quinoline nucleus is an important pharmacophore in medicinal chemistry, since a large number of its derivatives possess useful biological properties\(^2\text{-}^6\).

Pyrazoles are an important class of compounds in medicinal chemistry\(^7\text{-}^8\). It has been well documented that they possess antihypertensive\(^9\), antibacterial\(^10\), anti-inflammatory muscle relaxant\(^11\text{-}^12\) and antitumor properties\(^13\). The fused quinolines inhibit DNA topoisomerase and display cytotoxic and antitumor activities\(^14\). However, pyrazoloquinoline derivatives have been found to possess biological activities such as antiviral and antimalarial properties\(^15\text{-}^16\).

In the light of these findings, the synthesis of New Chemical Entities incorporating the pyrazoles and cyclohexanones may prove to be useful from the biological activity point of view.

Results and Discussion

2-Chloroquinoline-3-carboxaldehyde 1, on reaction with 4N HCl gave the previously reported the 3-formyl-2(1\(H\))-quinalone\(^17\) 2. Treatment of 2 with acetoephonone in 10% NaOH gave the corresponding chalcone derivative 3a (\(i.e., \) 3, \(Ar = Ph\)). The structure of 3a was determined on the basis of its spectral data. Thus, its IR (KBr) spectrum showed a broad and medium intensity band in the region 3001-2850 cm\(^{-1}\) assignable to –NH- stretching vibration, and strong, sharp peaks at 1674 and 1683 cm\(^{-1}\) assignable to the two carbonyl groups as the only diagnostic absorptions. Its \(^1\)H NMR spectrum (DMSO-\(d_6\)/TMS) showed signals at δ 7.23-8.35 (complex m, 11H, four aryl, five phenyl and two vinylic protons), 8.63 (s, 1H, quinoline peri proton), 12.11 (s, 1H, D\(_2\)O exchangeable –N\(H\)-). Its mass spectrum, when recorded in the CI method, showed the molecular ion (M\(^+\)\(+1\)) at \(m/z\) 276 corresponding to a molecular mass of 275. Condensation of 3a (\(i.e., \) 3, \(Ar = Ph\)) with phenylhydrazine in methanol containing catalytic amount of acetic acid under refluxing conditions for 30 minutes yielded the dihydro derivative 4a (\(i.e., \) 4, \(Ar = Ph\)). The structure of 4a was determined on the basis of its spectral data. Thus, its IR (KBr) spectrum showed a broad and medium intensity absorption in the region \(\approx3100\ cm^{-1}\) assignable to –NH- stretching vibration, and a strong, sharp absorption at 1655 cm\(^{-1}\) assignable to the carbonyl group as the only diagnostic peaks. Its \(^1\)H NMR spectrum (DMSO-
Further, reaction of 4a (i.e., 4, Ar = Ph) with chloranil in chloroform under reflux for 2 hr gave the dehydrogenated product i.e., 3-(2,5-diphenyl-2H-pyrazole-3-yl)-1H quinolin-2-one 5a (i.e., 5, Ar = Ph) whose structure was established on the basis of its spectral data. Thus, its IR (KBr) spectrum showed a broad and medium intensity peak at ≈3000 cm\(^{-1}\) assignable to –NH- stretching vibration, and a strong, sharp absorption at 1658 cm\(^{-1}\) assignable to the carbonyl group as the only diagnostic absorptions. Its \(^1\)H NMR spectrum (DMSO-d\(_6\)/TMS) showed signals at δ 6.74-7.75 (complex m, 16H, four aryl, 2 × five phenyl and quinalone peri protons), 12.08 (s, 1H, D\(_2\)O exchangeable–NH\(_2\)). Its mass spectrum, when recorded in the CI method, showed the molecular ion peak (M\(^+\)+1) at m/z 366 corresponding to a molecular mass of 365.

The compound thus obtained was found to be identical with 5a obtained above (3 → 4 → 5) in all respects such as m.p., m.m.p., co-TLC and IR.

Compound 3 on treatment with acetoacetanilide in the presence of catalytic amount of acetic acid in refluxing methanol for 30 min, yielded the corresponding quinalone-3-(cyclohexenone) 7a (i.e., 7, Ar = Ph). The structure of 7a was determined on the basis of its spectral data. Thus, its IR (KBr) spectrum showed a broad and medium intensity absorption in the region 3200-2800 cm\(^{-1}\) assignable to -NH- stretching vibration, and strong absorptions at 1645 and 1615 cm\(^{-1}\) assignable to the carbonyl groups. Its \(^1\)H NMR spectrum (DMSO-d\(_6\)/TMS) showed signals at δ 2.91 (two dd –CH\(_2\)H\(_2\) protons), 3.72 (m, –CH attached to two carbonyl groups), 6.72-7.78 (complex m, 16H, four aryl, 2 × five phenyl, one quinoline peri proton and one vinylic proton), 10.20 (s, 1H, D\(_2\)O exchangeable Ph–NH\(_2\)), 12.07 (s, 1H, D\(_2\)O exchangeable –NH\(_2\)). Its mass spectrum, when recorded in the CI method, showed the molecular ion peak (M\(^+\)+1) at m/z 435 corresponding to a molecular mass of 434. Treatment of 7a with chloranil in chloroform under refluxing conditions gave a dehydrogenated product 8a (i.e., 8, Ar = Ph) whose structure was confirmed on the basis of its spectral data (Scheme 1). Thus, its IR (KBr) spectrum showed a broad peak of medium intensity at 3100 cm\(^{-1}\) assignable to –NH- stretching vibration, and strong absorptions at 1650 and 1620 cm\(^{-1}\) assignable to the two carbonyl groups. Its \(^1\)H NMR spectrum showed signals at δ 4.64 (s, 1H, –CO–CH\(_2\)), 6.58-7.73 (complex m, 16H, four aryl, 2 × five phenyl and two protons), 7.91 (s, 1H, quinoline peri proton), 10.18 (s, 1H, D\(_2\)O exchangeable Ph–NH\(_2\)) and 11.88 (s, 1H, D\(_2\)O exchangeable quinoline –NH\(_2\)). Its mass spectrum, when recorded in the CI method, showed the molecular ion peak (M\(^+\)+1) at m/z 433 corresponding to a molecular mass of 432.

Experimental Section

Melting points are uncorrected and were determined in open capillary tubes in sulphuric acid bath. TLC checking was done on plastic sheets coated with silica gel GF-254, supplied by Merck and Co., and spotting was done using iodine vapour or UV lamp. IR spectra were recorded using Perkin-Elmer model 1000 instrument in KBr phase. \(^1\)H NMR spectra were recorded on a Gemini-200 and AU-400 operating at 200 MHz and 400 MHz respectively. Elemental analyses is given as % of C, H, N only for new compounds.

General procedure for the preparation of 3-(3-oxo-3-phenylpropenyl)-1H-quinolin-2-one 3

To a solution of 2 (1.73 g, 10 mmol) inaq. NaOH (10%, 30 mL) was added the respective
4-substituted ketone (10 mmol) at RT. The reaction mixture was stirred at RT for 2 hr. At the end of this period, the separated solid was filtered, washed with water (2 × 10 ml) and dried. The crude solid was purified by recrystallization from methanol to obtain pure 3.

3-(3-Oxo-3-phenylpropenyl)-1H-quinolin-2-one, 3a. Yield 2.5g (95%), m.p. 251°C (Lit18, m.p. 251°C). Spectral data is given under Results and Discussion Section.

3-[3-(4-Methoxyphenyl)-3-oxopropenyl]-1H-quinolin-2-one, 3b. Yield 2.7g (92%), m.p. 198°C; IR
(KBr): 3015-2855 (broad, medium,–NH–), 1650 cm⁻¹ (strong, sharp, carbonyl group); ¹H NMR (400 MHz, DMSO-d₆/TMS): δ 2.90-3.09 (s, 3H, –CH₃), 7.19-8.25 (complex m, 10H, four aryl, four phenyl and two vinylic protons), 8.73 (s, 1H, quinoline peri proton), 12.10 (s, 1H, D₂O exchangeable –NH–); MS: m/z 306 (M⁺+1). Anal. Found: C, 74.74; H, 4.95; N, 4.59%.

3-[3-(4-Nitrophenyl)-3-oxopropenyl]-1H-quinolin-2-one, 3c. Yield 2.8g (90%), m.p. >250°C; IR (KBr): 3010-2850 (broad, medium,–NH–), 8.60 (s, 1H, quinoline peri proton), 12.00 (s, 1H, D₂O exchangeable –NH–); MS: m/z 321 (M⁺+1). Anal. Found: C, 67.46; H, 4.88; N, 4.51. C₁₉H₁₈N₂O₄ requires: C, 74.67; H, 4.88; N, 4.51.

General procedure for the preparation of 3-(2,5-diphenyl-3,4-dihydro-2H-pyrazol-3-yl)-1H-quinolin-2-one, 4. A mixture of 3 (2.7 g, 10 mM), phenylhydrazine (1.98 mL, 20 mM) and methanol (15 mL) containing catalytic amount of acetic acid (2-3 drops) was refluxed for 30 min. At the end of this period, the separated solid was filtered, washed with cold methanol (2 x 5 mL) and dried. The crude solid was purified by recrystallization from methanol to obtain pure 4.

3-(2,5-Diphenyl-3,4-dihydro-2H-pyrazol-3-yl)-1H-quinolin-2-one, 4a. Yield 3.0g (86%), m.p. >250°C; Spectral data is given under Results and Discussion Section. Anal. Found: C, 78.75; H, 5.11; N, 11.38. C₂₂H₁₉N₂O₂ requires: C, 78.75; H, 5.11; N, 11.38.

3-[5-(4-Methoxy-phenyl)-2-phenyl-3,4-dihydro-2H-pyrazol-3-yl]-1H-quinolin-2-one, 4b. Yield 3.1g (80%), m.p. 198°C; IR (KBr): 3145-2805 (broad, medium,–NH–), 1665 cm⁻¹ (strong, sharp, –CO–); ¹H NMR (400 MHz, DMSO-d₆/TMS): δ 2.90-3.15 (s, 3H, –CH₃), 7.00 (d, 1H, another –C–H–), 7.36-8.12 (complex m, 8H, four aryl and four phenyl protons), 8.05 (s, 1H, quinoline peri proton), 12.00 (s, 1H, D₂O exchangeable –NH–); MS: m/z 396 (M⁺+1). Anal. Found: C, 75.88; H, 5.29; N, 10.58. C₂₅H₂₁N₂O₂ requires: C, 75.93; H, 5.35; N, 10.63%.

3-[5-(4-Nitrophenyl)-2-phenyl-3,4-dihydro-2H-pyrazol-3-yl]-1H-quinolin-2-one, 4c. Yield 3.2g (79%), m.p. >250°C; IR (KBr): 3160-2800 (broad, medium,–NH–), 1650 cm⁻¹ (strong, sharp, –CO–); ¹H NMR (400 MHz, DMSO-d₆/TMS): δ 3.11 and 3.91 (dd, pyrazole –CH₃H₂ protons), 5.47 (m,1H, pyrazole –CH– protons), 6.72-7.78 (complex m, 14H, four aryl, nine phenyl and quinoline peri protons), 12.07 (s, 1H, D₂O exchangeable –NH–); MS: m/z 411 (M⁺+1). Anal. Found: C, 70.18; H, 4.36; N, 13.59. C₂₃H₁₈N₄O₃ requires: C, 70.23; H, 4.42; N, 13.65%.

General Procedure for the preparation of 3-(1,2-Dibromo-3-oxo-3-phenyl-propyl)-1H-quinolin-2-one, 6. To a solution of 3 (2.7 g, 10 mM) in acetic acid (10 mL) was added a solution of bromine (0.53 mL, 10 mM) in acetic acid (5 mL) in a drop-wise manner with constant stirring at RT. After completion of addition, the mixture was stirred at RT for 8 hr. At the end of this period, the separated solid was filtered, washed with acetic acid (5 mL) and dried. The crude solid was purified by recrystallization from methanol to obtain pure 6.

3-(1,2-Dibromo-3-oxo-3-phenyl-propyl)-1H-quinolin-2-one, 6a. Yield 2.4g (58%), m.p. >250°C; Spectral data is given under Results and Discussion Section.

3-[1,2-Dibromo-3-(4-methoxy-phenyl)-3-oxo-propyl]-1H-quinolin-2-one, 6b. Yield 2.4g (56%), m.p. >250°C; IR (KBr): 3001-2850 (broad, medium,–NH–), 1670 cm⁻¹ (strong, sharp,–CO–); ¹H NMR (400 MHz, DMSO-d₆/TMS): δ 2.89-3.10 (s, 3H, –CH₃), 5.80 (d, 1H, –CH–), 7.00 (d, 1H, another –CH–), 7.25-8.20 (complex m, 8H, four aryl and four phenyl protons), 8.10 (s, 1H, quinoline peri proton), 12.00 (s, 1H, D₂O exchangeable –NH–).

3-[1,2-Dibromo-3-(4-nitrophenyl)-3-oxopropyl]-1H-quinolin-2-one, 6c. Yield 2.3g (51%), m.p. >250°C; IR (KBr): 3012-2870 (broad, medium,–NH–), 1665 cm⁻¹ (strong, sharp,–CO–); ¹H NMR (400 MHz, DMSO-d₆/TMS): δ 5.76 (d, 1H, –CH–), 6.85 (d, 1H, another –CH–), 7.36-8.12 (complex m, 8H, four aryl and four phenyl protons), 8.10 (s, 1H, quinoline peri proton), 11.67 (s, 1H, D₂O exchangeable –NH–).

General procedure for the preparation of 3-(2,5-diphenyl-3H-pyrazol-3-yl)-1H-quinolin-2-one, 5. A mixture of 4 (3.63 g, 10 mM), chloranil (2.45 g, 10 mM) and chloroform (40 mL) was refluxed for 2 hr. At the end of this period, the mixture was cooled to RT. The insoluble portion was filtered out and the chloroform solution was washed with 10% NaOH (50 mL) to free it from the 2,3,5,6-tetrachlorodihydroquinone. The organic layer was separated, washed with water...
(2×20 mL) until free of alkali and dried over anhyd. 
Na₂SO₄. The solvent was then distilled off to obtain a crude residue which was purified by recrystallization from methanol to obtain pure 5.

3-(2,5-Diphenyl-2H-pyrazol-3-yl)-1H-quinolin-2-one, 5a. Yield 2.9 g (82%); m.p.180°C; Spectral data is given under Results and Discussion Section. Anal. Found: C, 79.32; H, 4.72; N, 11.56%. C₂₉H₂₃N₃O requires: C, 76.28; H, 4.81; N, 10.62.

3-[5-(4-Methoxyphenyl)-2-phenyl-2H-pyrazol-3-yl]-1H-quinolin-2-one, 5b. Yield 3.1 g (81%), m.p.190°C; IR (KBr): 3109-2864 (broad, medium, –O–H), 1650 cm⁻¹ (strong, sharp, –CO–); ¹H NMR (400 MHz, DMSO-d₆/TMS): δ 2.94-3.02 (s, 3H, -CH₃), 6.84-7.75 (complex m, 15H, four aryl, nine phenyl, pyrazole -CH₃- and quinoline peri protons), 12.08 (s, 1H, D₂O exchangeable –NH₂); MS: m/z 394 (M⁺+1). Anal. Found: C, 76.28; H, 4.81; N, 10.62. C₂₉H₂₃N₃O₂ requires: C, 76.32; H, 4.87; N, 10.68%.

3-[5-(4-Nitrophenyl)-2-phenyl-2H-pyrazol-3-yl]-1H-quinolin-2-one, 5c. Yield 3.2 g (20%), m.p. 210°C; IR (KBr): 3100-2860 (broad, medium, –NH₂), 1650 cm⁻¹ (strong, sharp, -CO-); ¹H NMR (400 MHz, DMSO-d₆/TMS): δ 3.14, 3.92, 5.47 (m, 3 × 1H, pyrazole -CH₃- and -CH₃H₂- protons), 6.72-7.78 (complex m, 14H, four aryl, nine phenyl and quinoline peri protons), 12.07 (s, 1H, D₂O exchangeable –NH₂); MS: m/z 409 (M⁺+1). Anal. Found: C, 70.52; H, 3.88; N, 13.68. C₂₉H₁₈N₃O₃ requires: C, 70.58; H, 3.95; N, 13.72%.

Alternative preparation of 5 from 6

A mixture of 6 (4.35 g, 10 mM), phenylhydrazine (1.98 mL, 20 mM), methanol (15 mL) and catalytic amount of acetic acid (2-3 drops) was refluxed for 2-4 hr. At the end of this period, the reaction mixture was poured into ice-cold water. The separated solid was filtered, washed with water and dried to obtain crude, which was identical with 5 obtained above in all respects such as m.p, co-TLC and IR. Yield 2.2g (69%).

General procedure for the preparation of 2-oxo-6-(2-oxo-1,2-dihydroquinolin-3-yl)-4-phenylcyclohex-3-enecarboxylic acid phenylamide, 7

A mixture of 3 (2.75 g, 10 mM) acetoacanilide (1.77 g, 10 mM) and methanol (15 mL) and catalytic amount of acetic acid (2-3 drops) was refluxed for 30 min. At the end of this period, the separated solid was filtered, washed with cold methanol (2 × 5 mL) and dried. The crude solid was purified by recrystallization from methanol to obtain pure 7.

2-Oxo-6-(2-oxo-1,2-dihydroquinolin-3-yl)-4-phenylcyclohex-3-enecarboxylic acid phenylamide, 7a. Yield 3.5 g (82%); m.p. 210°C; Spectral data is given under Results and Discussion Section. Anal. Found: C, 77.29; H, 5.01; N, 6.31. C₂₉H₂₃N₃O₃ requires: C, 77.40; H, 5.01; N, 6.45%.

4-(4-Methoxyphenyl)-2-oxo-6-(2-oxo-1,2-dihydroquinolin-3-yl)-cyclohex-3-enecarboxylic acid phenylamide, 7b. Yield 3.9 g (85%), m.p. 201°C; IR (KBr): 3200-2820 (broad, medium, -NH₂), 1645 and 1620 cm⁻¹ (strong, sharp, -CO- groups); ¹H NMR (400 MHz, DMSO-d₆/TMS): δ 2.94-3.02 (s, 3H, -CH₃), 3.21 (two dd, 2H, -CH₂₃H₂-), 4.00 (m, 1H, -CH₂CH₂-), 4.44 (d, 1H, -CO-CH₂-), 6.60-7.85 (complex m, 14H, four aryl, nine phenyl and one vinyl proton), 8.00 (s, 1H, quinoline peri proton). 10.20 (s, 1H, D₂O exchangeable Ph–NH₂), 11.90 (s, 1H, D₂O exchangeable quinoline –NH₂); MS: m/z 465 (M⁺+1). Anal. Found: C, 74.92; H, 5.18; N, 6.00. C₂₉H₂₄N₃O₄ requires: C, 74.98; H, 5.21; N, 6.03%.

4-(4-Nitrophenyl)-2-oxo-6-(2-oxo-1,2-dihydroquinolin-3-yl)-cyclohex-3-enecarboxylic acid phenylamide, 7c. Yield 4.0g (82%), m.p. > 250°C; IR (KBr): 3012-2870 (broad, medium, -NH₂), 1645 and 1620 cm⁻¹ (strong, sharp, -CO- groups); ¹H NMR (400 MHz, DMSO-d₆/TMS): δ 3.15 (two dd, 2H, -CH₂₃H₂-), 3.95 (m, 1H, -CH₂CH₂-), 4.40 (d, 1H, -CO-CH₂-), 6.50-7.75 (complex m, 14H, four aryl, nine phenyl and one vinyl proton), 8.10 (s, 1H, quinoline peri proton), 10.10 (s, 1H, D₂O exchangeable Ph–NH₂), 11.80 (s, 1H, D₂O exchangeable quinoline –NH₂); MS: m/z 480 (M⁺+1). Anal. Found: C, 70.09; H, 4.36; N, 8.71. C₂₉H₂₃N₃O₃ requires: C, 70.14; H, 4.41; N, 8.76%.

General procedure for the preparation of 5-hydroxy-3-(2-oxo-1,2-dihydroquinolin-3-yl)-biphenyl-4-carboxylic acid phenylamide, 8

A mixture of 7 (4.35 g, 10 mM), chloranil (2.45 g, 10 mM) and chloroform (30 mL) was refluxed for 2 hr. At the end of this period, the mixture was cooled to RT. The insoluble portion (which is 2,3,5,6-tetrachloro-1,4-dihydroquinone) was filtered and the chloroform filtrate washed with 10% NaOH (50 mL). The organic layer was separated, washed with water (2 × 20 mL) free of alkali and dried over anhyd. Na₂SO₄ Then the solvent was distilled off to obtain a residue of crude 8. A portion of this solid was purified by recrystallization from chloroform-hexane to obtain the pure 8.
5-Hydroxy-3-(2-oxo-1,2-dihydro-quinolin-3-yl)-biphenyl-4-carboxylic acid phenylamide, 8a. Yield 3.2g (70%), m.p. 208°C; Spectral data is given under Results and Discussion Section. Anal. Found: C, 75.27; H, 4.57; N, 6.00. C_{28}H_{30}N_{2}O_{3} requires: C 75.31; H, 4.71; N, 6.06%.

5-Hydroxy-4'-methoxy-3-(2-oxo-1,2-dihydro-quinolin-3-yl)-biphenyl-4-carboxylic acid phenylamide, 8b. Yield 3.2g (70%), m.p. 225°C; IR (KBr): 3110-2864 (broad, medium, –NH-); 2929 (strong, sharp, –CO- groups); 1H NMR (400 MHz, DMSO-$_d_6$): δ 2.94-3.02 (s, 3H, –C$_d$H$_3$), 4.60 (s, 1H, –CO-CH$_2$–), 6.52-7.70 (complex m, 15H, four aryl, nine phenyl and two vinylic protons). 7.99 (s, 1H, quinoline peri proton), 10.25 (s, 1H, D$_2$O exchangeable Ph–NH$_2$–), 11.97 (s, 1H, D$_2$O exchangeable quinoline –NH–); MS: m/z 463 (M$^+$+1). Anal. Found: C, 75.31; H, 4.71; N, 6.06%.

5-Hydroxy-4'-nitro-3-(2-oxo-1,2-dihydro-quinolin-3-yl)-biphenyl-4-carboxylic acid phenylamide, 8c. Yield 3.3g (70%), m.p. 193°C; IR (KBr): 3110-2874 (broad, medium, –NH-), 1650 and 1620 cm$^{-1}$ (strong, sharp, –CO- groups); 1H NMR (400 MHz, DMSO-$_d_6$/TMS): δ 2.94-3.02 (s, 3H, –C$_d$H$_3$), 4.65 (s, 1H, -CO-C$_d$), 6.52-7.70 (complex m, 15H, four aryl, nine phenyl and two vinylic protons), 7.82 (s, 1H, quinoline peri proton), 10.15 (s, 1H, D$_2$O exchangeable Ph–NH$_2$–), 11.78 (s, 1H, D$_2$O exchangeable quinoline –NH–); MS: m/z 478 (M$^+$+1). Anal. Found: C, 70.31; H, 3.94; N, 8.76. C$_{28}$H$_{19}$N$_2$O$_4$ requires: C, 70.43; H, 4.01; N, 8.80%.

Conclusion

In conclusion, an efficient and general synthesis of quinalone-3-(N-phenylpyrazoles) 5 and quinalone-3-(cyclohexadienone) derivatives 8 as New Chemical Entities from quinalone-3-carboxaldehyde 2 have been described, which may posses useful biological properties.

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References