# Synthesis of 2-aminochromone-3-carbaldehyde hydrazones from 3-thiocarbamoylchromones and hydrazines

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Reaction of 3-thiocarbamoylchromones with hydrazines is accompanied by recyclization affording 2-aminochromone-3-carbaldehyde hydrazones.

**Key words:** heterocyclization, chromones, hydrazines, hydrazones, thioamides, recyclization.

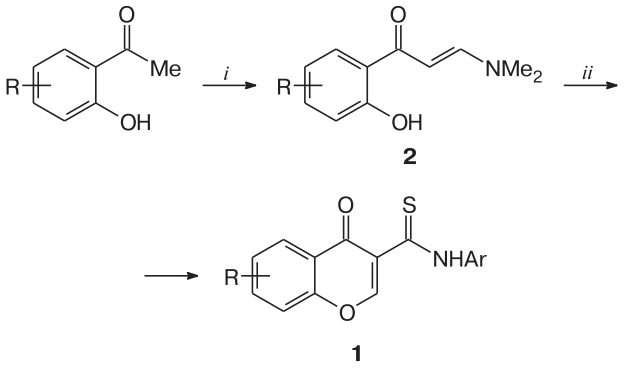
Chromone motif forms a part of a variety of natural and synthetic medicinal drugs[1—3] and photoactive products.[4,5] Moreover, chromones display a diverse reactivity. Of considerable synthetic potential are chromones 3-substituted with electron-withdrawing substituents, which enhance the push-pull activation of the pyrone C=C bond and stimulate its tendency to transformations by the action of nucleophiles. After nucleophilic addition to the double bond of the γ-pyrone ring, various recyclization "ring opening—ring closing" (RORC) reactions are typical for the resulting compounds. Reactivity of chromones bearing 3-positioned electron-withdrawing groups such as formyl, nitrile, ester, and carboxy, is well studied.[6,7] Carbamoylchromones[8] are studied to a lesser extent, while thiocarbamoyl derivatives are not studied at all due to the lack of a convenient synthetic approach thereto. At the same time the presence of thiocarbonyl moiety should effect significantly the direction of recyclizations occurring followed the nucleophilic attack, and consequently, the structure of the resulting products.

Earlier[9] we have developed a novel method to synthesize 3-thiocarbamoylchromones **1** based on the reaction between *o*-hydroxyaryl amino enones **2** and isothiocyanates (Scheme 1), allowing to start studying their transformation by the action of nucleophiles.

The aim of the present study was to investigate reactions of 3-thiocarbamoylchromones with various hydrazines.

We have shown that 3-thiocarbamoylchromones **1a—c** react with hydrazines **3a—c** in alcohol to produce 3-(R-hydrazono)methyl-2-arylamino-4*H*-chromen-4-ones **4a—j** (Scheme 2). The reaction comprises the consecutive processes such as the Michael reaction proceeding at the C(2) position of γ-pyrone ring (intermediate **A**), then the retro-Michael reaction with the pyrone ring opening (intermediate **B**), subsequent rotation around the C—C bond (intermediate **C**), and cyclization involving the hydroxyl group (intermediate **D**) to form a new pyrone ring (intermediate **E**) take place, and the final step is a rearrangement resulting in the formation of hydrazono aminochromones **4a—j**. It should be noted that the thiocarbonyl group is involved at the step of cyclization proceeding by the action of the hydroxy group to yield hydrazono aminochromones. There is no substitution of the aniline moiety in our case as occurs in reactions between nucleophiles and carbamoylchromones.

**Scheme 1**




**Reagents and conditions:** *i*. $Me_2NC(OMe)_2$, toluene, Δ. *ii*. ArNCS, DMF, Δ.

The structure of the resulting compounds was confirmed by the IR spectroscopy, mass spectrometry, and NMR spectroscopy data. In the IR spectra of the compounds, the C=O (1610 $cm^{-1}$), hydrazone C=N (1570 $cm^{-1}$) and the N—N bond (1440—1460 $cm^{-1}$) absorptions were

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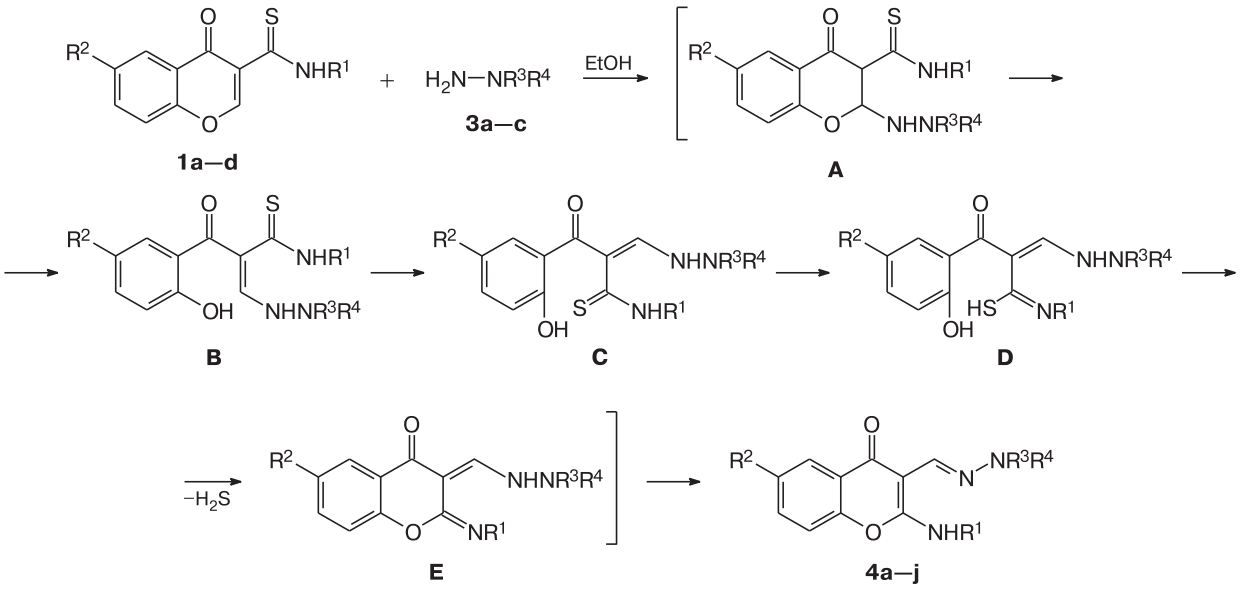
**Scheme 2**




| 1 | $R^1$ | $R^2$ |
|---|---|---|
| a | Ph | H |
| b | 4-$EtO_2CC_6H_4$ | H |
| c | 3,5-$Cl_2C_6H_3$ | H |
| d | Ph | F |

| 3 | $R^3$ | $R^4$ |
|---|---|---|
| a | Ph | H |
| b | $—(CH_2)_2—O—(CH_2)_2—$ | |
| c | $—(CH_2)_2—N(Me)—(CH_2)_2—$ | |

| 4 | $R^1$ | $R^2$ | $R^3$ | $R^4$ |
|---|---|---|---|---|
| a | Ph | H | Ph | H |
| b | Ph | H | $—(CH_2)_2—O—(CH_2)_2—$ | |
| c | Ph | H | $—(CH_2)_2—N(Me)—(CH_2)_2—$ | |
| d | 4-$EtO_2CC_6H_4$ | H | Ph | H |
| e | 4-$EtO_2CC_6H_4$ | H | $—(CH_2)_2—O—(CH_2)_2—$ | |
| f | 4-$EtO_2CC_6H_4$ | H | $—(CH_2)_2—N(Me)—(CH_2)_2—$ | |
| g | 3,5-$Cl_2C_6H_3$ | H | Ph | H |
| h | 3,5-$Cl_2C_6H_3$ | H | $—(CH_2)_2—O—(CH_2)_2—$ | |
| i | 3,5-$Cl_2C_6H_3$ | H | $—(CH_2)_2—N(Me)—(CH_2)_2—$ | |
| j | Ph | F | Ph | H |

observed. In mass spectra of the products, the parent peaks are accompanied with the peaks of comparable intensities ($[M - 92]^+$ for compounds **4a**,**d**,**g**,**j**, $[M - 86]^+$ for compounds **4b**,**e**,**h**, and $[M - 99]^+$ for compounds **4c**,**f**), which are supposedly formed upon the N—N bond cleavage in hydrazone moieties. Also, in all instances, peaks of 2-anilino-3-methylchromone and 2-hydroxyacetophenone were observed.

Structure of the products was unambiguously established by the example of compound **4j** using NMR spectroscopy wherein a complete assignement of all signals in $^1H$ and $^{13}C$ NMR spectra was carried out. Signals of phenyl protons as well as those of the chromone benzylidene ring appear as characteristic multiplets and were found at δ 6.7—7.8 corresponding to the area of aromatic protons. Three singlet peaks at δ 8.48, 10.37, and 11.78 are shifted downfield. The signals pertain to a proton on the $sp^2$-hybridized carbon atom and two NH groups, respectively. It is unclear from the one-dimensional $^1H$ NMR spectrum whether the CH moiety forms a part of a linear structure or a chromone heterocycle. Using a two-dimensional $^1H—^{13}C$ HSQC experiment, a correlation between the proton (δ 8.48) and the carbon atom having the chemical shift at δ 134.5, was revealed. Such chemical shift can not be attributed to C(2) and C(3) atoms, therefore the above CH moiety is a part of the linear structure. Consequently, 2- and 3-positions of chromone are substituted. From the $^1H—^1H$ COSY spectrum (Fig. 1), low-intensity correlations between NH-protons and *ortho*-protons on the phenyl rings were revealed testifying that benzene rings are bonded to the residie of the molecule through NH groups. Also, in the COSY spectrum, an interaction between one of the NH protons and the CH proton was found

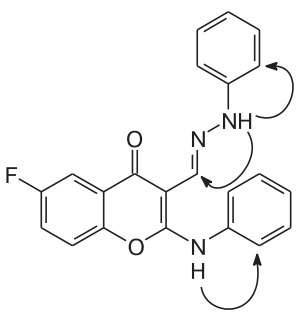


**Fig. 1.** Spin interaction found in a COSY experiment for compound **4j**.

($\delta_{NH}/\delta_{CH}$ = 10.37/8.48). Spin splitting was not observed in a one-dimensional spectrum, hence the COSY spectrum shows interactions over just four chemical bonds, and nitrogen atom is a linker. As a result, chromone is substituted at 2- and 3-positions with aniline and phenylhydrazonomethyl residues, respectively. Cross peaks being the evidence of this fact were found in a $^{1}H—^{13}C$ HMBC spectrum. Solution concentrations and time of 1D-experiments were chosen according to the known guidelines.[10]

To conclude, we have proposed a convenient procedure to obtain functionalized 2-aminochromone-3-carbaldehyde hydrazone derivatives. The reported[11,12] synthetic approach to hydrazones based on the reaction between hydrazines and 2-amino-3-formylchromones is more laborious and multi-step. It gave rise to the limited amount of hydrazones; 2-anilino-3-formylchromones were mostly used as starting compounds.

## Experimental

NMR spectra of compounds **4a—j** were recorded on a Bruker AM-300 spectrometer (300 ($^{1}H$) and 75 MHz ($^{13}C$)) in DMSO-$d_6$. Chemical shifts were measured relative to the deuterated solvent residual signals. IR spectrum were recorded on a Bruker ALPHA spectrometer in KBr pellets. Mass spectra were recorded on a Varian MAT CH-6 instrument with a direct sample injection into radiation source, ionization energy 70 eV, control voltage 1.75 kV. High resolution mass spectra (HRMS) were obtained on a Bruker micrOTOF II instrument using electrospray ionization technique. Melting points were measured on a Boetius apparatus and were uncorrected. The reaction was monitored by the TLC technique using TLC plates Merck 60 $F_{254}$ UV-254.

$^{1}H$ NMR and 2D-correlational spectra for compound **4j** were recorded on a Bruker Avance 600 spectrometer (600 ($^{1}H$) and 150 MHz ($^{13}C$)) in $CDCl_3$ at 293 K. $^{13}C$ NMR spectra were obtained on a Bruker DRX500 spectrometer (125 MHz) in $CDCl_3$ at room temperature. The residual solvent signals were used as an internal standard. Two-dimensional correlation spectrum was obtained using the standard Bruker software.

**Synthesis of 3-thiocarbamoylchromones 1a—d (general procedure).** A mixture of amino enone **2** (15 mmol) and a corresponding isothiocyanate (33 mmol, 2.2 equiv.) in dried DMF (3 mL) was heated for 3—5 h at 110 °C (TLC monitoring). The solvent was evaporated and the product was purified on a silica gel column (eluent — $CH_2Cl_2$).

**Synthesis of 2-amino-3-formylchromone hydrazones 4a—j (general procedure).** A mixture of hydrazine **3a—c** (0.5 mmol) and thiocarbamoylchromone **1a—d** (0.5 mmol) in alcohol (5 mL) was stirred for 3 h at room temperature (for phenylhydrazine) or heated for 2 h at 60 °C (for aliphatic hydrazines). The precipitate was filtered and recrystallized from ethyl acetate (for phenylhydrazine) or rinsed with alcohol (for aliphatic hydrazines).

**2-Phenylamino-3-[(2-phenylhydrazono)methyl]-4*H*-chromen-4-one (4a).** Yield 35%, yellow powder, m.p. 155—157 °C. IR, $\nu/cm^{-1}$: 3433, 3231, 3041, 1654, 1622, 1600, 1567, 1542, 1498, 1465, 1434, 1331, 1299, 1264, 1228, 1215, 1159, 1102, 1026, 994, 943, 897, 754, 686. $^{1}H$ NMR (300 MHz, DMSO-$d_6$), δ: 11.74 (s, 1 H); 10.21 (s, 1 H); 8.55 (s, 1 H); 8.08 (d, 1 H, *J* = 7.6 Hz); 7.70 (t, 1 H, *J* = 7.5 Hz); 7.65—7.39 (m, 6 H, *J* = 24.0 Hz, *J* = 17.2 Hz, *J* = 7.5 Hz); 7.34—7.20 (m, 3 H, *J* = 16.2 Hz, *J* = 8.0 Hz); 6.94 (d, 2 H, *J* = 7.8 Hz); 6.77 (t, 1 H, *J* = 7.2 Hz). $^{13}C$ NMR (75 MHz, DMSO-$d_6$), δ: 173.26, 157.60, 154.60, 152.80, 145.55, 136.70, 135.37, 133.42, 130.14, 129.78, 125.75, 125.61, 125.44, 122.38, 119.03, 117.60, 112.02, 95.10. HRMS, found: *m/z* 356.1405 $[M]^+$. Calculated for $C_{22}H_{17}N_3O_2$: M = = 356.1394.

**3-[(Morpholinoimino)methyl]-2-phenylamino-4*H*-chromen-4-one (4b).** Yield 47%, yellowish powder, m.p. 159—161 °C. IR, $\nu/cm^{-1}$: 3491, 3011, 2973, 2938, 2879, 2825, 1649, 1604, 1559, 1469, 1445, 1370, 1333, 1274, 1223, 1187, 1114, 1092, 1004, 947, 932, 865, 820, 759, 720, 665. $^{1}H$ NMR (300 MHz, DMSO-$d_6$), δ: 12.29 (s, 1 H); 8.26 (s, 1 H); 8.10—8.02 (m, 1 H); 7.70 (dd, 1 H, *J* = 11.2 Hz, *J* = 4.3 Hz); 7.55 (d, 3 H, *J* = 6.1 Hz); 7.46 (dd, 3 H, *J* = 14.2 Hz, *J* = 7.1 Hz); 7.25 (t, 1 H, *J* = 7.3 Hz); 3.86—3.76 (m, 4 H); 3.15—3.07 (m, 4 H). $^{13}C$ NMR (75 MHz, DMSO-$d_6$), δ: 173.28, 157.97, 152.85, 136.89, 134.85, 133.47, 130.01, 125.78, 125.44, 125.38, 122.50, 121.98, 117.62, 94.64, 66.04, 52.62. HRMS, found: *m/z* 350.1502 $[M]^+$. Calculated for $C_{20}H_{19}N_3O_3$: M = 350.1499.

**3-[(4-Methylpiperazin-1-yl)imino]methyl-2-phenylamino-4*H*-chromen-4-one (4c).** Yield 53%, white powder, m.p. 178—180 °C. IR, $\nu/cm^{-1}$: 3483, 3016, 2946, 2881, 2841, 2826, 2796, 2762, 2662, 1655, 1622, 1571, 1499, 1466, 1446, 1375, 1335, 1290, 1279, 1228, 1216, 1143, 1102, 1076, 1028, 1000, 937, 899, 834, 794, 750, 699, 688, 669. $^{1}H$ NMR (300 MHz, DMSO-$d_6$), δ: 12.27 (s, 1 H); 8.19 (s, 1 H); 8.05 (d, 1 H, *J* = 6.9 Hz); 7.72—7.62 (m, 1 H); 7.48 (d, 6 H, *J* = 13.5 Hz); 7.27—7.18 (m, 1 H); 3.10 (s, 4 H); 2.61 (s, 4 H); 2.30 (s, 3 H). $^{13}C$ NMR (75 MHz, DMSO-$d_6$), δ: 173.24, 157.84, 152.78, 136.91, 134.65, 133.36, 129.98, 125.70, 125.41, 125.28, 122.48, 121.85, 117.53, 94.74, 54.07, 51.45, 45.58. HRMS, found: *m/z* 363.1816 $[M]^+$. Calculated for $C_{21}H_{22}N_4O_2$: M = 363.1816.

**Ethyl 4-{[4-oxo-3-(2-phenylhydrazonomethyl)-4*H*-chromen-2-yl]amino}benzoate (4d).** Yield 37%, yellow powder, m.p. 221—223 °C. IR, $\nu/cm^{-1}$: 3431, 3249, 3039, 2979, 2903, 1718, 1655, 1603, 1561, 1541, 1509, 1493, 1466, 1425, 1365, 1333, 1300, 1275, 1229, 1182, 1106, 1025, 944, 902, 845, 751, 690. $^{1}H$ NMR (300 MHz, DMSO-$d_6$), δ: 11.85 (s, 1 H); 10.19 (s, 1 H); 8.50 (s, 1 H); 8.07 (d, 3 H, *J* = 6.6 Hz); 7.76—7.56 (m, 4 H, *J* = 22.8 Hz); 7.45 (t, 1 H, *J* = 7.5 Hz); 7.26 (t, 2 H, *J* = 6.1 Hz); 6.95 (d, 2 H, *J* = 6.1 Hz); 6.78 (t, 1 H, *J* = 7.6 Hz); 4.34 (q, 2 H, *J* = 6.6 Hz); 1.36 (t, 3 H, *J* = 5.6 Hz). $^{13}C$ NMR (75 MHz, DMSO-$d_6$), δ: 173.52, 165.55, 156.97, 152.81, 145.36, 141.17, 134.79, 133.61, 131.31, 129.82, 125.92, 125.43, 122.29, 121.36, 121.21, 119.21, 117.78, 112.14, 96.03, 61.12, 14.62. HRMS, found: *m/z* 428.1595 $[M]^+$. Calculated for $C_{25}H_{21}N_3O_4$: M = 428.1605.

**Ethyl 4-[(3-morpholinoiminomethyl-4-oxo-4*H*-chromen-2-yl)amino]benzoate (4e).** Yield 46%, yellowish powder, m.p. 181—183 °C. IR, $\nu/cm^{-1}$: 3434, 2976, 2840, 1718, 1655, 1607, 1561, 1510, 1459, 1434, 1408, 1365, 1307, 1277, 1233, 1180, 1107, 1007, 901, 845, 762, 701, 673. $^{1}H$ NMR (300 MHz, DMSO-$d_6$), δ: 12.43 (s, 1 H); 8.18 (s, 1 H); 8.08—7.94 (m, 3 H); 7.76—7.53 (m, 4 H); 7.48—7.37 (m, 1 H); 4.33 (q, 2 H, *J* = 6.3 Hz); 3.80 (s, 4 H); 3.29 (s, 4 H); 1.34 (t, 3 H, *J* = 6.0 Hz). $^{13}C$ NMR (75 MHz, DMSO-$d_6$), δ: 173.47, 165.52, 157.30, 152.78, 141.39, 134.10, 133.54, 131.10, 125.85, 125.63, 125.40, 122.38, 120.70, 117.69, 95.52, 65.97, 61.05, 52.40, 14.60. HRMS, found: *m/z* 422.1715 $[M]^+$. Calculated for $C_{23}H_{23}N_3O_5$: M = 422.1710.

**Ethyl 4-({3-[(4-methylpiperazin-1-yl)iminomethyl]-4-oxo-4*H*-chromen-2-yl}amino)benzoate (4f).** Yield 52%, white powder, m.p. 140—142 °C. IR, $\nu/cm^{-1}$: 3449, 2986, 2938, 2881, 2825,

2803, 1709, 1657, 1625, 1604, 1567, 1511, 1459, 1432, 1408, 1364, 1281, 1231, 1215, 1181, 1138, 1108, 1027, 1002, 937, 899, 843, 796, 765, 701, 673, 628. $^1$H NMR (300 MHz, DMSO-$d_6$), δ: 12.45 (s, 1 H); 8.08 (s, 1 H); 8.05—7.92 (m, 3 H, $J$ = 14.0 Hz, $J$ = 8.1 Hz); 7.68 (t, 1 H, $J$ = 7.3 Hz); 7.56 (d, 3 H, $J$ = 7.9 Hz); 7.41 (t, 1 H, $J$ = 7.2 Hz); 4.31 (dd, 2 H, $J$ = 14.0 Hz, $J$ = 7.0 Hz); 3.11 (s, 4 H); 2.59 (s, 4 H); 2.29 (s, 3 H); 1.34 (t, 3 H, $J$ = 7.0 Hz). $^{13}$C NMR (75 MHz, DMSO-$d_6$), δ: 173.41, 165.50, 157.14, 152.71, 141.38, 133.86, 133.44, 131.07, 125.77, 125.49, 125.36, 122.34, 120.50, 117.61, 95.59, 61.03, 53.98, 51.24, 45.58, 14.58. HRMS, found: $m/z$ 435.2022 $[M]^+$. Calculated for $C_{24}H_{26}N_4O_4$: M = 435.2027.

**2-(3,5-Dichlorophenylamino)-3-(2-phenylhydrazonomethyl)-4*H*-chromen-4-one (4g).** Yield 34%, yellow powder, m.p. 238—240 °C. IR, ν/cm$^{-1}$: 3425, 3273, 1655, 1628, 1598, 1587, 1561, 1542, 1509, 1497, 1465, 1449, 1421, 1329, 1299, 1266, 1226, 1194, 1159, 1117, 1040, 993, 955, 927, 843, 809, 749, 735, 685, 670. $^1$H NMR (300 MHz, DMSO-$d_6$), δ: 11.80 (s, 1 H); 10.15 (s, 1 H); 8.49 (s, 1 H); 8.08 (d, 1 H, $J$ = 7.2 Hz); 7.80—7.67 (m, 1 H); 7.61 (s, 2 H); 7.54—7.38 (m, 3 H); 7.31—7.17 (m, 2 H); 6.96 (d, 2 H, $J$ = 6.3 Hz); 6.80 (s, 1 H). $^{13}$C NMR spectrum could not be obtained because of poor solubility of the substance. HRMS, found: $m/z$ 424.0615 $[M]^+$. Calculated for $C_{22}H_{15}Cl_2N_3O_2$: M = 424.0614.

**2-(3,5-Dichlorophenylamino)-3-morpholinoiminomethyl-4*H*-chromen-4-one (4h).** Yield 49%, yellowish powder, m.p. 176—178 °C. IR, ν/cm$^{-1}$: 3441, 3232, 2951, 2924, 2869, 2826, 1656, 1625, 1585, 1561, 1542, 1465, 1432, 1372, 1320, 1272, 1211, 1142, 1116, 1063, 1005, 955, 926, 890, 839, 803, 755, 721, 699, 662, 602. $^1$H NMR (300 MHz, DMSO-$d_6$), δ: 12.14 (s, 1 H); 8.21 (s, 1 H); 8.04 (s, 1 H); 7.71 (s, 1 H); 7.57—7.33 (m, 5 H); 3.81 (s, 4 H); 3.30 (s, 4 H). $^{13}$C NMR spectrum could not be obtained because of poor solubility of the substance. HRMS, found: $m/z$ 418.0713 $[M]^+$. Calculated for $C_{20}H_{17}Cl_2N_3O_3$: M = 418.0720.

**2-(3,5-Dichlorophenylamino)-3-[(4-methylpiperazin-1-yl)-iminomethyl]-4*H*-chromen-4-one (4i).** Yield 50%, white powder, m.p. 196—198 °C. IR, ν/cm$^{-1}$: 3448, 3233, 3024, 2941, 2875, 2823, 2800, 2768, 1665, 1624, 1587, 1560, 1465, 1434, 1375, 1326, 1290, 1254, 1215, 1145, 1110, 1077, 1002, 957, 925, 887, 837, 806, 759, 698, 671, 604. $^1$H NMR (300 MHz, DMSO-$d_6$), δ: 12.19 (s, 1 H); 8.15 (s, 1 H); 8.04 (d, 1 H, $J$ = 7.3 Hz); 7.71 (t, 1 H, $J$ = 7.2 Hz); 7.44 (dd, 5 H, $J$ = 27.0 Hz, $J$ = 18.3 Hz); 3.28 (s, 4 H); 2.55 (s, 4 H); 2.26 (s, 3 H). $^{13}$C NMR spectrum could not be recorded because of poor solubility of the substance. HRMS, found: $m/z$ 431.1042 $[M]^+$. Calculated for $C_{21}H_{20}Cl_2N_4O_2$: M = 431.1008.

**6-Fluoro-2-phenylamino-3-[(2-phenylhydrazono)methyl]-4*H*-chromen-4-one (4j).** Yield 37%, yellow powder, m.p. 142—144 °C. IR, ν/cm$^{-1}$: 3412, 3226, 3040, 1674, 1615, 1599, 1558, 1540, 1499, 1457, 1432, 1331, 1289, 1257, 1226, 1207, 1160, 1100, 1010, 989, 941, 885, 750, 646. $^1$H NMR (400 MHz, DMSO-$d_6$), δ: 11.78 (s, 1 H, NH); 10.37 (s, 1 H, NHN); 8.48 (s, 1 H, CHN); 7.71 (dd, 1 H, H(5), $J$ = 8.4 Hz, $J$ = 3.1 Hz); 7.64 (dd, 1 H, H(8), $J$ = 9.1 Hz, $J$ = 4.3 Hz); 7.61—7.55 (m, 3 H, *o′*-H, H(7)); 7.52 (t, 2 H, *m′*-H, $J$ = 7.9 Hz); 7.29 (t, 1 H, *p′*-H, $J$ = 7.4 Hz); 7.24 (t, 2 H, *m*-H, $J$ = 7.9 Hz); 6.90 (d, 2 H, *o*-H, $J$ = 7.4 Hz); 6.76 (t, 1 H, *p*-H, $J$ = 7.3 Hz). $^{13}$C NMR (100 MHz, DMSO-$d_6$), δ: 172.0 (C(4)), 159.1 (d, C(6), $J$ = 243.4 Hz), 157.3 (C(2)), 148.6 (C(8a)), 145.0 (*i*-C), 136.1 (*i′*-C), 134.5 (CHN), 129.8 (*m′*-C), 129.4 (*m*-C), 125.4 (*p′*-C), 123.3 (d, C(4a), $J$ = 7.1 Hz), 122.1 (*o′*-C), 120.6 (d, C(7), $J$ = 25.2 Hz), 119.9 (d, C(8), $J$ = 8.5 Hz), 118.8 (*p*-C), 111.6 (*o*-C), 110.0 (d, C(5), $J$ = 24.0 Hz), 94.5 (C(3)). HRMS, found: $m/z$ 374.1406 $[M]^+$. Calculated for $C_{22}H_{16}FN_3O_2$: M = 374.1401.

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