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SYNTHESIS OF (*E*)-2,2-DIMETHYLCHROMAN-4-ONE-O-((1-BENZYL-1H-1,2,3-TRIAZOL-4-YL)METHYL)OXI MES AND (E)-2,2-DIMETHYLCHROMAN-4-ONE-O-((1-PHENYL-1H-1,2,3-TRIAZOL-4-YL)METHYL)OXIMES

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Abstract:
A new series of (*E*)-2,2-dimethylchroman-4-one-O-((1-benzyl-1H-1,2,3-triazol-4-yl)methyl)oxi mes and (*E*)-2,2-dimethylchroman-4-one-O-((1-phenyl-1H-1,2,3-triazol-4-yl)methyl)oximes were synthesized by the reaction of (*E*)-2,2-dimethylchroman-4-one-O-prop-2-yn-1-yloxime and substituted benzyl/phenyl azides.

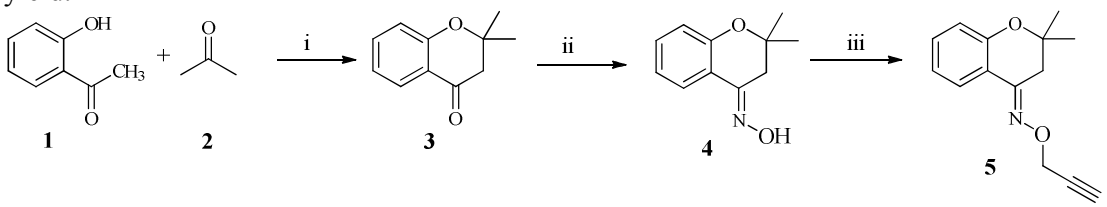
Keywords: Heterocycles, triazoles, chromanone

Introduction:

Triazoles, which are an important heterocyclic compounds, have been studied for over a century because of their broad range of biological activities.[i-iv] More recently, there has been significant interest in the development of novel triazoles with anti-inflammatory, antiplatelet, antimicrobial, antimycobacterial, antitumoral, and antiviral properties and activity against several neglected diseases.[v]

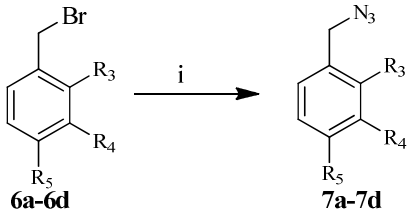
Result and discussion:

The reaction of 2-hydroxy acetophenone (**1**) with acetone (**2**) in the presence of piperidine resulted in 2,2-dimethylchroman-4-one[vi] (**3**). These chromanone on reaction with hydroxylamine hydrochloride gave oxime[vii] (**4**). It was subjected to alkylation with propargylbromide gave (*E*)-2,2-dimethylchroman-4-one-O-prop-2-yn-1-yloxime (**5**) in good yield.[viii]



Scheme-1: Reagents and conditions: i) Piperidine, EtOH, reflux, 10 h ii) Hydroxylaminehydro chloride, EtOH iii) Propargyl bromide, KO*t*Bu, THF

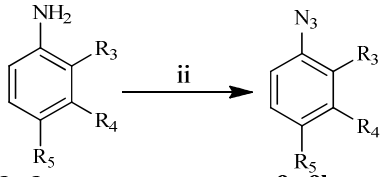
Benzyl bromides (**6a-6d**) on reaction with sodium azide in DMF solvent led to benzyl azides (**7a-7d**) and anilines (**8e-8k**) dissolved in con HCl reacted with $NaNO_2$ followed by reaction with NaN_3 furnish phenyl azides[ix] (**9e-9k**).



6a-6d **7a-7d**

a= R_3=R_4=R_5=H
b= R_3=R_4=H, R_5=Cl
c= R_3=R_5=Cl, R_4=H
d= R_3=H, R_4=R_5=Cl

Scheme-2: Reagents and conditions: NaN_3, DMF, rt, 6 h

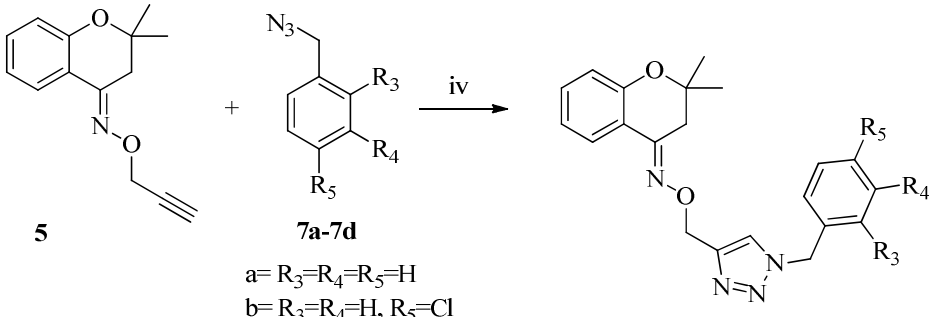


8e-8k **9e-9k**

e=R_3=R_4=R_5=H i=R_3=R_5=H R_4=Cl
f=R_3=R_4=H, R_5=Br j=R_3=R_5=H R_4=OCH_3
g=R_3=R_4=H, R_5=Cl k=R_3=Cl, R_4=R_5=H
h=R_3=R_4=H, R_5=F

Scheme-3: Reagents and conditions: Con HCl, $NaNO_2$, NaN_3

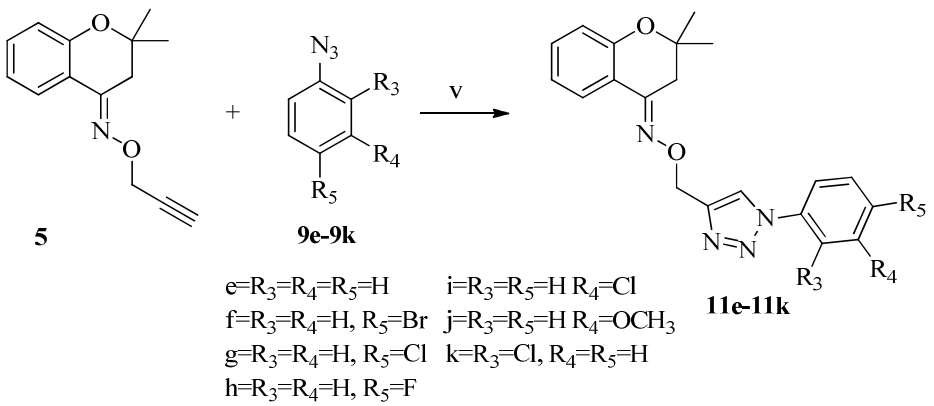
Reaction of (*E*)-2,2-dimethylchroman-4-one-O-prop-2-yn-1-yloxime (**5**) with substituted benzyl azides (**7a-7d**) led to (*E*)-2,2-dimethylchroman-4-one-O-((1-benzyl-1H-1,2,3-triazol-4-yl)methyl)oximes (**10a-10d**) and with phenyl azides (**9e-9k**) to form (*E*)-2,2-dimethylchroman-4-one-O-((1-phenyl-1H-1,2,3-triazol-4-yl)methyl)oximes (**11e-11k**).



5 **7a-7d** **10a-10d**

a= R_3=R_4=R_5=H
b= R_3=R_4=H, R_5=Cl
c= R_3=R_5=Cl, R_4=H
d= R_3=H, R_4=R_5=Cl

Scheme-4: Reagents and conditions: iv. $CuSO_4.5H_2O$, Sodium ascorbate, DMF



Scheme-5: Reagents and conditions: v. $CuSO_4.5H_2O$, Sodium ascorbate, DMF

Conclusion:

Novel triazole derivatives comprising chromanone moiety were synthesized and characterized.

Acknowledgements:

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Experimental section:

General remarks

Air or/and moisture sensitive reactions were carried out in anhydrous solvents under an argon atmosphere in an oven or flame-dried glassware. All anhydrous solvents were distilled prior to use: THF from Na and benzophenone; CH_2Cl_2, DMSO from CaH_2. Commercial reagents were used without purification. Column chromatography was carried out by using ACME silica gel (60-120 mesh).

Infrared spectra were recorded on Perkin-Elmer 683 spectrometer. 1H and 13C NMR spectra were recorded on a Varian Unity-500 or Varian INOVA 400 MHz or a Bruker-300 spectrometer. Chemical shifts (δ) are given in ppm relative to TMS and coupling constants (J) in Hz. The following abbreviations are used to designate signal multiplicity: s = singlet, d = doublet, dd = doublet of doublet, t = triplet, td = triplet of doublet, q = quartet, m = multiplet, br = broad. Mass spectra were obtained on an Exactive Thermo Scientific Orbit rap Mass Spectrometer.

Synthesis of 2,2-dimethylchroman-4-one (3):

To a well stirred solution of *o*-hydroxyacetophenone (5 g, 36.7 mmol) and propan-2- one (2.35 g, 40.4 mmol) in ethanol (30 mL), pyrrolidine (3.13 g, 44.1 mmol) was added at room temperature. The reaction mixture was refluxed for 3 h. The completion of the reaction was monitored by TLC. The reaction mixture was quenched by crushed ice and extracted with ethyl acetate. The organic layer was washed by brine (2×25 mL) and dried over anhydrous sodium sulfate, and the solvent was removed under pressure to obtain the crude product. The obtained residues were purified by column chromatography on silica gel, ethylacetate and pet ether as eluent to give product **3** 6.08 g, 94% yield.

Physical state: white solid. m.p: 85-87 ^oC. IR: 2977, 2932, 1690, 1603, 1573, 1456, 1372, 1329, 1304, 1257, 1202, 1169, 1117, 768 cm^{-1}. ^1H-NMR (500 MHz, $CDCl_3$): δ 7.84 (dd, *J*=1.5 Hz, 1.5 Hz H-7), 7.44 (t, H-5), 6.94 (t, H-6), 6.90 (d, *J*=8.3 Hz, H-8), 2.72 (s, 3-CH_2), 1.46 (s, 6H) ppm. ^{13}C-NMR (75 MHz, $CDCl_3$): δ 192.4, 159.8, 136.0, 126.3, 120.5, 120.0, 118.2, 79.0, 48.7, 26.5 ppm. ESI-MS: 177 $[M+H]^+$.

Synthesis of (E)-2,2-dimethylchroman-4-one oxime (4)

Potassium carbonate (1.56 g, 11.35 mmol) and hydroxylamine hydrochloride (788 mg, 11.35 mmol) were added to a stirred solution of 3,4-dihydro-2,2-dimethyl-2*H*-1-benzopyran-4-one (1 g, 5.675 mmol) in ethanol (15 ml). The suspension was refluxed for 3 h. The reaction was then poured onto ice and, after complete melting of ice, water was added to obtain a final volume of 195 ml. The title compound was collected by filtration, washed with water and dried (950 mg, 87 %).

Physical state: White powder; M.P: 123-125 °C; IR: (KBr): υ 3264, 2977, 2935, 1650, 1260, 931 cm^{-1}; ^{1}H-NMR (400 MHz): (DMSO-d_6, 500 MHz): δ 1.30 (s, 6H), 2.77 (s, 2H), 6.82 (d, J = 8.2 Hz, 1H), 6.89 (t, J = 7.7 Hz, 1H), 7.24 (t, J = 7.8 Hz, 1H), 7.74 (d, J = 7.8 Hz, 1H), 11.19 (s, 1H). ^{13}C-NMR (400 MHz): 157.2, 147.9, 131.6, 131.3, 120.4, 117.3, 114.5, 83.8, 34.2, 27.7 ESI-MS: 192 $[M+H]^+$.

Synthesis of (*E*)-2,2-dimethylchroman-4-one O-prop-2-yn-1-yl oxime (5)

KO*t*Bu (645 mg, 5.75 mmol) was added to a solution of benzaldehyde oxime (1.0 g, 5.229 mmol) in THF (15 mL) at 0 ^{o}C. After 15 min propargyl bromide (0.59 mL, 7.843 mmol) was added dropwise and stirred overnight before the addition of NH_4Cl (aq) (20 mL). The resultant mixture was extracted with EtOAc, dried, and concentrated in vacuo. Purification by column chromatography gave **5** (1.02 g, 85%) as a clear yellow oil.

Physical state: yellow oil; IR: 3293, 3264, 2925, 1635, 1528, 1483, 1456, 1379, 1303, 1243, 1188, 1033, 753 Cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 7.58 (t, *J* = 6.4 Hz, 1H), 7.41 (m, 1H), 7.25 (t, 1H), 6.96 (d, 1H), 4.25 (d, *J* = 2.4 Hz, 2H), 3.32 (t, *J* = 2.4 Hz, 1H), 3.06 (s, 2H), 1.46 (s, 3H), 1.34 (s, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): 157.1, 151.3, 131.4, 131.1, 120.4, 117.3, 114.5, 83.6, 78.7, 76.3, 61.2, 34.5, 27.7 ppm; ESI-MS: 230 $[M+H]^+$.

Synthesis of (*E*)-2,2-dimethylchroman-4-one O-((1-benzyl-1H-1,2,3-triazol-4-yl)methyl)oxi me (10a):

Physical state: colorless solid; M.P: 188-190 °C; IR: 3290, 3093, 2922, 2872, 2358, 2338, 1619, 1596, 1573, 1470, 1292, 1268, 1216, 1132, 1110, 1078 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 7.63 (s, 1H), 7.58 (m, 1H), 7.41 (m, 1H), 7.33 (m,2H), 7.25 (m,2H), 7.23 (m, 2H), 6.98 (m,1H), 5.47 (s, 2H), 4.76 (s, 2H), 3.05 (s, 2H), 1.46 (s, 3H), 1.35 (s, 3H) ppm; ^{13}C-NMR (400 MHz, $CDCl_3$): 157.4, 151.3, 142.4, 133.7, 131.4, 128.6, 127.6, 125.7, 122.6, 120.3, 117.3, 114.5, 83.6, 67.5, 57.3, 34.5, 27.4 ppm; ESI-MS: 385 $[M+Na]^+$.

Synthesis of (E)-2,2-dimethylchroman-4-one O-((1-(4-chlorobenzyl)-1H-1,2,3-triazol-4-yl)methyl)oxime (10b):

Physical state: yellow solid; M.P: 215-217 °C; IR: 3278, 3090, 2920, 2870, 2356, 2334, 1614, 1590, 1577, 1470, 1286, 1264, 1215, 1131, 1106, 1075 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 7.63 (s, 1H), 7.58 (m, 1H), 7.47 (m,1H), 7.37 (m, 2H), 7.26 (m, 1H), 7.17 (m, 2H), 6.98 (m, 1H), 5.48 (s,2H), 4.79 (s, 2H), 3.06 (s, 2H), 1.46 (s, 3H), 1.35 (s,3H) ppm; ^{13}C-NMR (400 MHz):157.2, 151.4, 142.3, 134.3, 131.6, 131.3, 128.7, 122.9, 120.3, 117.1, 114.5, 83.7, 67.5, 57.4, 34.4, 27.5 ppm; ESI-MS: 397 $[M+H]^+$, 399 $[M+H+2]^+$.

(*E*)-2,2-dimethylchroman-4-one O-((1-(2,4-dichlorobenzyl)-1H-1,2,3-triazol-4-yl)methyl) oxime (10c):

Physical state: brown solid; M.P: 192-194 ^{o}C; IR: 3282, 3094, 2922, 2875, 2354, 2337, 1619, 1593, 1573, 1474, 1289, 1262, 1217, 1135, 1108, 1072 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 7.66 (d, J=6.4 Hz, 1H), 7.62 (s, 1H), 7.58 (m,1H), 7.41(m, 1H), 7.24 (m,2H), 7.11 (d, J=6.3 Hz, 1H), 6.98 (m, 1H), 5.48 (s, 2H), 4.75 (s, 2H), 3.04 (s, 2 H), 1.44 (s, 3H), 1.35 (s, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): 157.7, 151.4, 142.1, 136.1, 135.9, 132.4, 131.5, 131.2, 130.2, 126.6, 122.7, 120.4, 117.0, 114.3, 83.4, 67.5, 49.9, 34.3, 27.7 ppm; ESI-MS: 432

$[M+H]^+$, 434 $[M+H+2]^+$. **(E)-2,2-dimethylchroman-4-oneO-((1-(3,4-dichlorobenzyl)-1H-1,2,3-triazol-4-yl)methyl)oxi me (10d):**

Physical state: brown solid; M.P: 174-176 ^oC; IR: 3280, 3092, 2920, 2871, 2352, 2334, 1616, 1591, 1572, 1479, 1284, 1257, 1220, 1128, 1103, 1069 cm^{-1}; ^1H-NMR (400 MHz, $CDCl_3$): 7.63 (s, 1H), 7.61 (d, J= 8.2 Hz, 1H), 7.58 (m, 1H), 7.41 (d, J= 7.2 Hz, 1H), 7.37 (t, J=7.5 Hz, 1H), 7.35 (d, J=7.7 Hz, 1H), 7.04 (d, J= 8.2 Hz, 1H), 6.98 (t, J=7.8 Hz, 1H), 5.48 (s, 2H), 4.79 (s, 2H), 3.05 (s, 2H), 1.46 (s,3H), 1.35 (s,3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): 157.4, 151.6, 142.4, 135.8, 131.6, 131.3, 130.6, 130.2, 126.7, 122.7, 120.4, 117.2, 114.4, 83.7, 67.5, 56.8, 34.5, 27.7 ppm; ESI-MS: 432 $[M+H]^+$, 434 $[M+H+2]^+$.

(E)-2,2-dimethylchroman-4-one O-((1-phenyl-1H-1,2,3-triazol-4-yl)methyl) oxime (11e):

Physical state: brown liquid; IR: 3280, 3135, 3093, 2997, 2918, 2877, 2364, 1639, 1617, 1594, 1523, 1490, 1319, 1284, 1192, 1114, 1089, 1051, 1032, 1007 cm^{-1}; ^1H-NMR (400 MHz, $CDCl_3$): 8.08 (s, 1H), 7.62-7.58 (m, 5H), 7.45-7.41 (m, 2H), 7.26 (m, 1H), 6.98 (m, 1H), 4.79 (d, J=12.2 Hz, 2 H), 3.06 (s, 2H), 1.45 (s, 3H), 1.35 (s, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): 157.6, 151.4, 144.4, 136.7, 131.6, 131.3, 128.6, 120.5, 120.4, 119.1, 117.3, 114.4, 83.6, 67.3, 34.5, 27.8 ppm; ESI-MS: 349 $[M+H]^+$.

(E)-2,2-dimethylchroman-4-oneO-((1-(4-bromophenyl)-1H-1,2,3-triazol-4-yl)methyl) oxime (11f):

Physical state: pale yellow solid; M.P:256-258 ^oC; IR: 3277, 3129, 3091, 2994, 2914, 2871, 2360, 1636, 1611, 1598, 1527, 1485, 1316, 1278, 1188, 1110, 1088, 1048, 1030, 1004 cm^{-1}; ^1H-NMR (400 MHz, $CDCl_3$): 8.08 (s, 1H), 7.60- 7.51 (m, 5H), 7.41 (m, 1H), 7.26 (m, 1H), 6.98 (m, 1H), 4.79 (s, 2H), 3.04 (s, 2H), 1.45 (s, 3H), 1.35 (s, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): 157.0, 151.3, 144.6, 135.9, 131.6, 131.3, 128.0, 123.2, 120.4, 119.1, 117.3, 114.6, 83.6, 67.5, 34.6, 27.8 ppm; ESI-MS: 428 $[M+H]^+$, 430 $[M+H+2]^+$.

(E)-2,2-dimethylchroman-4-one O-((1-(4-chlorophenyl)-1H-1,2,3-triazol-4-yl)methyl)oxime (11g):

Physical state: colorless solid; M.P: 167-169 ^oC; IR: 3269, 3127, 3090, 2992, 2913, 2869, 2364, 1631, 1609, 1596, 1524, 1488, 1312, 1272, 1189, 1107, 1083, 1040, 1028, 1000 cm^{-1}; ^1H-NMR (400 MHz, $CDCl_3$): 8.06 (s, 1H), 7.58-7.41 (m,6H), 7.25 (m, 1H), 6.97 (m, 1H), 4.77 (s, 2H), 3.07 (s, 2H), 1.45 (s, 3H), 1.35 (s, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): 157.4, 151.2, 144.4, 135.6, 131.8, 131.5, 128.2, 123.0, 120.2, 119.3, 117.0, 114.3, 83.5, 67.5, 34.8, 27.6 ppm; ESI-MS: 383 $[M+H]^+$, 385 $[M+H+2]^+$.

(E)-2,2-dimethylchroman-4-one O-((1-(4-fluorophenyl)-1H-1,2,3-triazol-4-yl)methyl)oxime (11h):

Physical state: light red solid; M.P:223-225 ^oC; IR: 3271, 3131, 3093, 2993, 2913, 2864, 2369, 1629, 1604, 1595, 1522, 1485, 1310, 1268, 1179, 1103, 1079, 1044, 1024, 998 cm^{-1}; ^1H-NMR (400 MHz, $CDCl_3$): 8.09 (s, 1H), 7.60-7.58 (m, 3H), 7.41 (m,1H), 7.26-7.24 (m, 3H), 6.97 (m, 1H), 4.79 (s, 2H), 3.06 (s, 2H), 1.45 (s, 3H), 1.35 (s, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): 162.8, 157.3, 151.4, 144.4, 132.6, 131.8, 122.5, 120.4, 119.1, 117.2, 115.4, 114.3, 83.8, 67.5, 34.8, 27.6 ppm; ESI-MS: 367 $[M+H]^+$.

(E)-2,2-dimethylchroman-4-one O-((1-(3-chlorophenyl)-1H-1,2,3-triazol-4-yl)methyl) oxime (11i):

Physical state: brown liquid; IR: 3268, 3133, 3093, 2990, 2909, 2862, 2365, 1626, 1600, 1587, 1520, 1482, 1306, 1266, 1175, 1107, 1074, 1042, 1020, 994 cm^{-1}; ^1H-NMR (400 MHz, $CDCl_3$): 8.08 (s, 1H), 7.81 (s, 1H), 7.58- 7.39 (m, 5H), 7.26 (m, 1H), 6.98 (m, 1H), 4.79 (s, 2H), 3.04 (s, 2H), 1.46 (s, 3H), 1.33 (s, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): 157.3, 147.6, 139.7, 135.1, 131.8, 131.5, 130.7, 122.3, 120.5, 117.3, 117.1, 114.3, 95.0, 83.5, 34.8, 27.6 ppm; ESI-MS: 383 $[M+H]^+$, 385 $[M+H+2]^+$.

(E)-2,2-dimethylchroman-4-oneO-((1-(3-methoxyphenyl)-1H-1,2,3-triazol-4-yl)methyl)oxi me (11j):

Physical state: white solid; M.P: 260-262 ^{o}C; IR: 3274, 3132, 3090, 2994, 2919, 2872, 2367, 1635, 1619, 1595, 1528, 1492, 1315, 1288, 1190, 1112, 1086, 1050, 1028, 1003 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 8.08 (s, 1H), 7.58 (s, 1H), 7.41- 7.34 (m, 2H), 7.26-7.18 (m, 2H), 6.99- 6.95 (m, 3H), 4.79 (s, 2H), 3.83 (s, 3H), 3.04 (s, 2H), 1.46 (s, 3H), 1.33 (s, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): 162.6, 157.3, 151.3, 144.6, 142.7, 131.8, 131.5, 129.7, 122.0, 120.5, 119.1, 117.1, 114.3, 108.3, 83.5, 67.5, 34.8, 27.6 ppm; ESI-MS: 379 $[M+H]^+$.

(E)-2,2-dimethylchroman-4-one O-((1-(2-chlorophenyl)-1H-1,2,3-triazol-4-yl)methyl) oxime (11k):

Physical state: brown solid; M.P: 196-198 ^{o}C; IR: 3274, 3122, 3094, 2992, 2903, 2865, 2364, 1631, 1604, 1593, 1520, 1484, 1310, 1270, 1185, 1109, 1078, 1036, 1024, 998 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 8.06 (s, 1H), 7.58-7.39 (m, 5H), 7.26 (m, 1H), 6.97 (m, 1H), 4.77 (s, 2H), 3.07 (s, 2H), 1.46 (s, 3H), 1.35 (s, 3H) ppm; ^{13}C-NMR (100 MHz): 157.3, 151.4, 144.5, 132.7, 132.1, 131.8, 131.5, 130.7, 129.8, 128.8, 120.5, 119.1, 117.3, 114.3, 83.5, 67.5, 34.5, 27.6 ppm; ESI-MS: 383 $[M+H]^+$, 385 $[M+H+2]^+$.

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