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ARTICLE *in* HETEROATOM CHEMISTRY · JANUARY 2005

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Heteroatom Chemistry
Volume 16, Number 6, 2005

Synthesis of 3-Substituted 2-Trifluoro(trichloro)methyl-2*H*-chromenes by Reaction of Salicylaldehydes with Activated Trihalomethyl Alkenes

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Received 20 March 2005

ABSTRACT: *The reaction of activated trihalomethylsubstituted alkenes with salicylaldehydes in the presence of triethylamine gives 3-substituted 2-trifluoromethylchroman-4-ols and 2-trifluoro(trichloro)-methyl-2H-chromenes in high yields.* © 2005 Wiley Periodicals, Inc. Heteroatom Chem 16:492–496, 2005; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20146

INTRODUCTION

Substituted 2,2-dimethylchromans and chromenes are common natural products which are widely distributed among many plants [1]. Furthermore, they have considerable biological importance, especially as potentially useful pesticides (antijuvenile hormones precocene I and precocene II [2,3]) and drug candidates in the field of potassium channel openers (for example, cromakalim, a highly potent antihypertensive drug [4–7]). Several analogues of 2-methyl-2*H*-chromene also show interesting pharmaceutical activity and are potential medicinal agents [1,8]. Because of their reactivity and relative stability, 2-methyl- and 2,2-dimethyl-2*H*-chromenes have an important role and are valuable intermediates for synthetic purposes in chroman chemistry [9–14].

The introduction of fluorine in place of hydrogen to modify the bioactivity of organic molecules is a well-established practice [15–18]. As a result, considerable efforts have been made in the development of trifluoromethylated analogues of precocenes [19–21], cromakalim [22–24], and lactarochromal [19,20,25], in which both or one of the methyl groups in the *gem*-dimethyl moiety are replaced by the CF_3 group. In spite of advances in this area, published data on the synthesis of 2-(trihalomethyl)-2*H*-chromenes are lacking. To our knowledge, there has been only one report on the preparation of 2-(trifluoromethyl)chroman-4-one [26], which may be regarded as a precursor for the synthesis of 2-trifluoromethyl-2*H*-chromene.

RESULTS AND DISCUSSION

It is well known that the reactions of salicylaldehydes with acrylonitrile [27–29], alkyl vinyl ketones [30–33], and nitro alkenes [34–39] give Δ^3-chromenes containing electron-withdrawing substituents at the 3-position. Although these compounds are not found in nature, their derivatives are reported to be useful

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Contract grant sponsor: Russian Foundation for Basic Research.
Contract grant number: 04-03-32463.

from industrial and medical points of view [40,41]. These facts and our continuing interest in the chemistry of trifluoromethyl analogues of natural chromans and chromenes [19,20,25] led us to investigate the synthesis of the hitherto unknown 2-CX_3-3-R-2*H*-chromenes (X = F, Cl; R = COPh, NO_2). In this work, we report a simple and convenient synthesis of these compounds involving the condensation of salicylaldehydes **1** with trihaloethylidene derivatives of acetophenone and nitromethane **2a–c**, prepared from trifluoro(trichloro)acetaldehyde hydrates [42–45]. Although much attention has been paid to the chemistry of alkenes **2** mainly due to the possibility of using them as an excellent building blocks for the preparation of a variety of CX_3-containing compounds [46–53], their reactions with salicylaldehydes were not described in the literature.

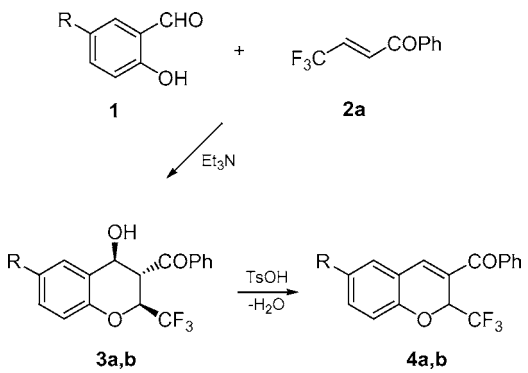
We have found that salicylaldehyde and 5-bromosalicylaldehyde react with (*E*)-4,4,4-trifluoro-1-phenyl-2-buten-1-one (**2a**) in the presence of triethylamine in dichloromethane for 1–3 days at room temperature to afford chromanols **3a,b** in 71% and 56% yields, respectively. In all cases, only one regio- and stereo-isomer was obtained (Scheme 1). A plausible mechanism for the reaction involves triethylamine catalyzed tandem conjugate addition/aldol-type reaction [38]. When triethylamine was replaced by DABCO, the reaction did not occur and only resinification was observed.

The configuration and the conformational preferences of chromanols **3a,b** have been assigned on the basis of ^{1}H NMR data. In particular, two coupling constants, the $J_{2,3}$ = 10.5–10.6 Hz (axial–axial) and $J_{3,4}$ = 9.7 Hz (axial–pseudoaxial), indicate an equatorial position for the 2-CF_3 and 3-COPh substituents and a pseudoequatorial position for the 4-OH group in the mobile dihydropyran fragment of **3a,b**, which are therefore *trans-trans* products [34]. The subsequent dehydration of the chromanols **3a,b** to the corresponding 2*H*-chromenes **4a,b** was performed in refluxing toluene for 3 h in the presence of *p*-toluene sulfonic acid as a catalyst in excellent yields (Scheme 1).

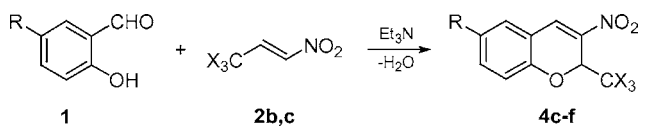
Next, we investigated the reaction of salicylaldehydes **1** with (*E*)-3,3,3-trifluoro- and 3,3,3-trichloro-1-nitroprop-1-enes (**2b,c**), for this, it was anticipated, would give a range of new trihalomethylated 3-nitro-2*H*-chromenes as precursors to a variety of medicinally important chroman derivatives [40,41]. It turned out that unlike alkene **2a**, the reaction of trihaloethylidene nitromethanes **2b,c** with salicylaldehydes **1** is much faster, reaching completion within 10 min to 2 h. Moreover, under the conditions used, additional step to affect dehydration of the corresponding chromanols **3** was not necessary since the reaction proceeded smoothly to give 2-trifluoromethyl- and 2-trichloromethyl-3-nitro-2*H*-chromenes **4c–f** directly in 76–99% yields (Scheme 2). It seems that the CX_3 group favors the initial Michael addition reaction due to its electron-withdrawing character, which lowers the LUMO level of the molecule [54]. However, the halogenated substituents do not exercise significant control over the reaction: the regiochemistry is determined by the nitro or acyl group. In the light of the present interest in fluoro-containing compounds as pharmaceutical intermediates [15–18], this novel entry to fluorinated analogues of 2-methyl-2*H*-chromene is noteworthy.

The structures of compounds **4** compare well with the results of elemental analysis, ^{1}H, ^{19}F NMR, and IR spectroscopy. A characteristic feature of the ^{1}H NMR spectra of **4c–f** is the appearance of singlet at δ 8.03–8.13 ppm for the H-4 proton and quartet at δ 6.09 ppm with $J_{H,F}$ = 6.2–6.3 Hz for the H-2 proton (singlet at δ 6.32–6.33 ppm in the case of **4e,f**). All reactions are clean, easy to perform, and proceed at room temperature; however, trichloroethylidene acetonitrile did not react under similar reaction conditions. The results obtained by using two salicylaldehydes **1** and three activated alkenes **2a–c** are summarized in Table 1.

In conclusion, the reaction of salycilaldehydes with activated trihalomethyl substituted alkenes provides convenient preparative process from readily available starting materials to 2-CF_3- and 2-CCl_3-2*H*-chromenes, which may be considered as a new precursors in the synthesis of other useful chroman derivatives.



SCHEME 1



SCHEME 2

TABLE 1 Synthesis of Chromanols **3a,b** and Chromenes **4a–f** by Reaction of Salicylaldehydes **1** with Trihalomethyl Substituted Alkenes **2a–c**

R	*X*	*Compound*	*Yield (%)*	*Mp (°C)*
H	F	**3a**	71	195–196
Br	F	**3b**	56	205–206
H	F	**4a**	98	98–100
Br	F	**4b**	80	110–112
H	F	**4c**	76	82–83
Br	F	**4d**	93	97–98
H	Cl	**4e**	94	105–106
Br	Cl	**4f**	85	123–125

EXPERIMENTAL

Melting points obtained were uncorrected. IR spectra were recorded on an Perkin-Elmer Spectrum BX-II instrument as KBr disks. ^{1}H and ^{19}F NMR spectra were recorded on a Bruker DRX-400 spectrometer (^{1}H at 400 MHz and ^{19}F at 376 MHz) with TMS and C_6F_6 as internal standards. All solvents used were dried and distilled per standard procedures. The starting trihalomethyl substituted alkenes **2a–c** were prepared by direct condensation of the appropriate trihaloacetaldehyde hydrates with acetophenone and nitromethane according to described procedures [42–45].

Trans-trans-3-Benzoyl-2-(trifluoromethyl)chroman-4-ol (**3a**). To a solution of salicylaldehyde (0.64 g, 5.2 mmol) and alkene **2a** (1.05 g, 5.2 mmol) in dichloromethane (10 mL) was added triethylamine (0.10 g, 1.0 mmol). The mixture was allowed to stand for 72 h at r.t. After partial evaporation of the solvent, the residue was diluted with hexane (6 mL) and the crystalline material was collected by filtration to give 1.2 g (71%) of **3a** as a colorless powder. IR (KBr): ν 3485, 3420, 1668, 1583, 1485 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$/TMS): δ 2.27 (d, 1H, OH, J = 7.4 Hz), 4.15 (dd, 1H, H-3, J = 10.5, 9.7 Hz), 4.90 (dq, 1H, H-2, J = 10.5, 5.8 Hz), 5.30 (dd, 1H, H-4, J = 9.7, 7.4 Hz), 6.99 (dd, 1H, H-8, J = 8.2, 1.0 Hz), 7.08 (td, 1H, H-6, J = 7.5, 1.0 Hz), 7.29 (br t, 1H, H-7, J = 7.8 Hz), 7.45-7.55 (m, 3H, H-5, H-3′, H-5′), 7.64 (tt, 1H, H-4′, J = 7.4, 1.0 Hz), 8.01–8.04 (m, 2H, H-2′, H-6′). ^{19}F NMR (376 MHz, $CDCl_3/C_6F_6$): δ 85.63 (d, CF_3, J = 5.8 Hz). Anal. Calcd for $C_{17}H_{13}F_3O_3$: C, 63.36; H, 4.07. Found: C, 63.00; H, 3.95.

Trans-trans-3-Benzoyl-6-bromo-2-(trifluoromethyl)chroman-4-ol (**3b**). This compound was prepared analogously to **3a** for 24 h as a colorless powder. IR (KBr): ν 3483, 3431, 1673, 1633, 1475 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$/TMS): δ 2.32 (d, 1H, OH, J = 7.2 Hz), 4.12 (dd, 1H, H-3, J = 10.6, 9.7 Hz), 4.90 (dq, 1H, H-2, J = 10.6, 5.7 Hz), 5.27 (dd, 1H, H-4, J = 9.7, 7.2 Hz), 6.88 (d, 1H, H-8, J = 8.7 Hz), 7.38 (ddd, 1H, H-7, J = 8.7, 2.4, 1.0 Hz), 7.50–7.55 (m, 2H, H-3′, H-5′), 7.63 (dd, 1H, H-5, J = 2.4, 0.8 Hz), 7.65 (tt, 1H, H-4′, J = 7.4, 1.1 Hz), 8.00–8.03 (m, 2H, H-2′, H-6′). ^{19}F NMR (376 MHz, $CDCl_3/C_6F_6$): δ 85.58 (d, CF_3, J = 5.7 Hz). Anal. Calcd for $C_{17}H_{12}BrF_3O_3$: C, 50.90; H, 3.01. Found: C, 51.03; H, 3.01.

3-Benzoyl-2-(trifluoromethyl)-2H-chromene (**4a**). A mixture of **3a** (0.11 g, 0.3 mmol) and a catalytic amounts of TsOH in toluene (5 mL) was refluxed for 3 h. The resulting solution was concentrated under reduced pressure, and the precipitate that formed was recrystallized from hexane as a colorless powder. IR (KBr): ν 1637, 1603, 1571, 1482 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$/TMS): δ 6.09 (q, 1H, H-2, J = 7.1 Hz), 6.99 (td, 1H, H-6, J = 7.5, 1.0 Hz), 7.02 (d, 1H, H-8, J = 8.2 Hz), 7.17 (dd, 1H, H-5, J = 7.5, 1.6 Hz), 7.29 (s, 1H, H-4), 7.35 (ddd, 1H, H-7, J = 8.2, 7.5, 1.6 Hz), 7.50–7.55 (m, 2H, H-3′, H-5′), 7.62 (tt, 1H, H-4′, J = 7.4, 1.3 Hz), 7.75–7.78 (m, 2H, H-2′, H-6′). ^{19}F NMR (376 MHz, $CDCl_3/C_6F_6$): δ 82.76 (d, CF_3, J = 7.1 Hz). Anal. Calcd for $C_{17}H_{11}F_3O_2$: C, 67.11; H, 3.64. Found: C, 67.10; H, 3.63.

3-Benzoyl-6-bromo-2-(trifluoromethyl)-2H-chromene (**4b**). This compound was prepared analogously to **4a** as a colorless powder. IR (KBr): ν 1645, 1598, 1564, 1473 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$/TMS): δ 6.09 (q, 1H, H-2, J = 7.0 Hz), 6.92 (d, 1H, H-8, J = 8.7 Hz), 7.20 (s, 1H, H-4), 7.32 (d, 1H, H-5, J = 2.4 Hz), 7.43 (dd, 1H, H-7, J = 8.7, 2.4 Hz), 7.50–7.55 (m, 2H, H-3′, H-5′), 7.64 (tt, 1H, H-4′, J = 7.5, 1.3 Hz), 7.74–7.77 (m, 2H, H-2′, H-6′). ^{19}F NMR (376 MHz, $CDCl_3/C_6F_6$): δ 83.03 (d, CF_3, J = 7.0 Hz). Anal. Calcd for $C_{17}H_{10}BrF_3O_2$: C, 53.29; H, 2.63. Found: C, 53.43; H, 2.60.

General Procedure for the Synthesis of 3-Nitrochromenes **4c–f**

To a solution of salicylaldehyde **1** (10 mmol) and nitroalkene **2** (10 mmol) in a minimal volume of dichloromethane (1.5–5 mL) was added triethylamine (0.15 g, 1.5 mmol). The mixture was stirred for 2 h (10 min in the case of **4d**) at r.t. After evaporation of the solvent, the residue was recrystallized from hexane to give compound **4** as yellow needles.

3-Nitro-2-(trifluoromethyl)-2H-chromene (**4c**). IR (KBr): ν 1651, 1608, 1571, 1525, 1457, 1327 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$/TMS): δ 6.09 (q, 1H, H-2, J = 6.3 Hz), 7.07 (d, 1H, H-8, J = 8.2 Hz), 7.11 (td, 1H, H-6, J = 7.5, 1.0 Hz), 7.37 (dd, 1H, H-5, J = 7.6, 1.6 Hz), 7.46 (ddd, 1H, H-7, J = 8.2, 7.5, 1.6 Hz), 8.12 (s, 1H, H-4). ^{19}F NMR (376 MHz, $CDCl_3/C_6F_6$): δ 83.92

(d, CF_3, $J = 6.3$ Hz). Anal. Calcd for $C_{10}H_6F_3NO_3$: C, 48.99; H, 2.47; N, 5.71. Found: C, 49.03; H, 2.49; N, 5.61.

6-Bromo-3-nitro-2-(trifluoromethyl)-2H-chromene (**4d**). IR (KBr): ν 1650, 1599, 1564, 1520, 1473, 1331 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$/TMS): δ 6.09 (q, 1H, H-2, $J = 6.2$ Hz), 6.98 (d, 1H, H-8, $J = 8.7$ Hz), 7.50 (d, 1H, H-5, $J = 2.4$ Hz), 7.54 (dd, 1H, H-7, $J = 8.7$, 2.4 Hz), 8.04 (s, 1H, H-4). ^{19}F NMR (376 MHz, $CDCl_3/C_6F_6$): δ 84.02 (d, CF_3, $J = 6.2$ Hz). Anal. Calcd for $C_{10}H_5BrF_3NO_3$: C, 37.07; H, 1.56; N, 4.32. Found: C, 37.16; H, 1.45; N, 4.24.

3-Nitro-2-(trichloromethyl)-2H-chromene (**4e**). IR (KBr): ν 1642, 1605, 1526, 1453, 1328 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$/TMS): δ 6.33 (s, 1H, H-2), 7.09 (td, 1H, H-6, $J = 7.5$, 1.0 Hz), 7.11 (d, 1H, H-8, $J = 8.2$ Hz), 7.36 (dd, 1H, H-5, $J = 7.5$, 1.6 Hz), 7.46 (ddd, 1H, H-7, $J = 8.2$, 7.5, 1.6 Hz), 8.13 (s, 1H, H-4). Anal. Calcd for $C_{10}H_6Cl_3NO_3$: C, 40.78; H, 2.05; N, 4.76. Found: C, 40.76; H, 1.87; N, 4.75.

6-Bromo-3-nitro-2-(trichloromethyl)-2H-chromene (**4f**). IR (KBr): ν 1642, 1601, 1562, 1523, 1472, 1335 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$/TMS): δ 6.32 (s, 1H, H-2), 7.01 (dd, 1H, H-8, $J = 8.7$, 0.4 Hz), 7.49 (d, 1H, H-5, $J = 2.4$ Hz), 7.54 (dd, 1H, H-7, $J = 8.7$, 2.4 Hz), 8.03 (s, 1H, H-4). Anal. Calcd for $C_{10}H_5BrCl_3NO_3$: C, 32.17; H, 1.35; N, 3.75. Found: C, 32.34; H, 1.25; N, 3.72.

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