Journal of Organometallic Chemistry 902 (2019) 120971

Contents lists available at ScienceDirect

Journal of Organometallic Chemistry

journal homepage: www.elsevier.com/locate/jorganchem

Stereoselective synthesis of trisubstituted alkenyl Fischer aminocarbenes through self-mediated α-haloketone olefination

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ARTICLE INFO

Article history:
Received 2 September 2019
Received in revised form
8 October 2019
Accepted 9 October 2019
Available online 11 October 2019

Keywords:
Trisubstituted alkenes
Fischer aminocarbene complexes
Olefination
Stereoselective synthesis
α-Bromoketones

1. Introduction

Tri- and tetrasubstituted double bonds are frequently occurring motifs in nature. Shikimic acid [1], α-tocotrienol [2] and lycophyll [3] are examples of naturally occurring trisubstituted alkenes, whereas jasmine [4], kitol [5] and β-damascenone [6] are examples of tetrasubstituted alkenes. Trisubstituted [7] and tetrasubstituted [8] alkenes are associated with interesting biological activities. Moreover, alkenes have found many applications in organic synthesis [9]. It is not surprising that the synthesis of tri- [10] and tetrasubstituted [11] alkenes has gained considerable attention in the last couple of decades.

Fischer carbene complexes form a large group of substances that have significantly contributed to the field of organic synthesis [12]. Among the Fischer carbenes, alkenylated Fischer aminocarbenes bearing disubtituted or trisubstituted double bond are interesting substrates given their reactivity and availability. Aminocarbene complexes with disubstituted double bonds are easily prepared through condensation with aldehydes [13] and through the exchange reaction of α,β-unsaturated alkoxy carbene complexes with amines [14]. However, the reaction of α,β-unsaturated amides with $Na_2Cr(CO)_5$ [15] has a limited scope. The synthetic applications of the abovementioned carbene complexes include their use in benzannulations [16], Diels–Alder [14] and cycloaddition reactions [17], double bond reduction [18], metal exchange studies [19] and SET-H atom transfer process [20]; they are also used as nucleophiles in alkylation reactions [21] and in addition-elimination reactions [22] or as acceptor in NHC addition reaction [23]. By contrast, trisubstituted alkenyl Fischer aminocarbene complexes have fewer synthetic applications, such as in metal exchange study [24], nucleophilic addition [25], benzannulation reaction [26] and ring closure [27]. The availability of these complexes is limited to several examples that are prepared via Diels–Alder reaction [28], cross-metathesis reaction [29], ynamine insertion [30], amine addition to allenylidene complexes [31], and exchange reaction of α,β-unsaturated alkoxy carbene complexes with amines [32], among others [33].

2. Results and discussion

In our research on the preparation and reactivity of functionalized Fischer carbene complexes [34], we observed that treatment of an excess of aminocarbene **1a** with butyllithium (BuLi) followed by reaction with phenacyl bromide led to the stereoselective formation of (*E*)-trisubstituted alkene bearing dimethylaminocarbene moiety with a yield of 18% (Table 1, entry 1). This phenomenon attracted our attention; thus, we optimized the reaction conditions to determine the reliable and simple procedure to prepare functionalized trisubstituted alkenyl aminocarbene complexes. The use of 1.3 equiv of the carbene **1a** increased the yield of **3a** to 63% (Table 1, entry 2). A mixture of THF and ether as reaction solvents reduced the equivalents of the complex **1a**, maintaining the isolated yield of **3a** at 63% (Table 1, entry 3). The yield of **3a** significantly improved when a mixture of tetrahydrofurane–toluene was used. By contrast, the same reaction in a mixture of tetrahydrofurane–acetonitrile substantially decreased the yield of the trisubstituted alkene **3a** (Table 1, entries 4,5). An attempt to perform the condensation reaction in dry toluene resulted in no reaction (Table 1, entry 6). Unreacted starting carbene complex **1a**

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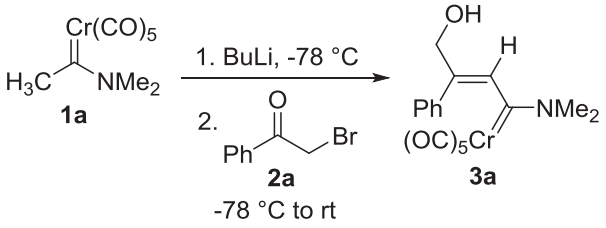
https://doi.org/10.1016/j.jorganchem.2019.120971

Table 1
Optimization of trisubstituted alkene **3a** formation.



Entry	2a, Equiv	Time (h)	Solvent	3a[%][a]
1	**2a**, 1.1	3	THF	18
2	**2a**, 1.3	3	THF	63
3	**2a**, 1.1	3	THF/Et_2O	63
4	**2a**, 1.1	3	THF/toluene	80 (64%[b])
5	**2a**, 1.1	3	THF/MeCN	51
6	**2a**, 1.1	4	toluene	–[c]

[a] Isolated yield.
[b] The reaction was performed in a 4 mmol scale.
[c] The unreacted carbene complex **1a** remained in the crude reaction mixture.

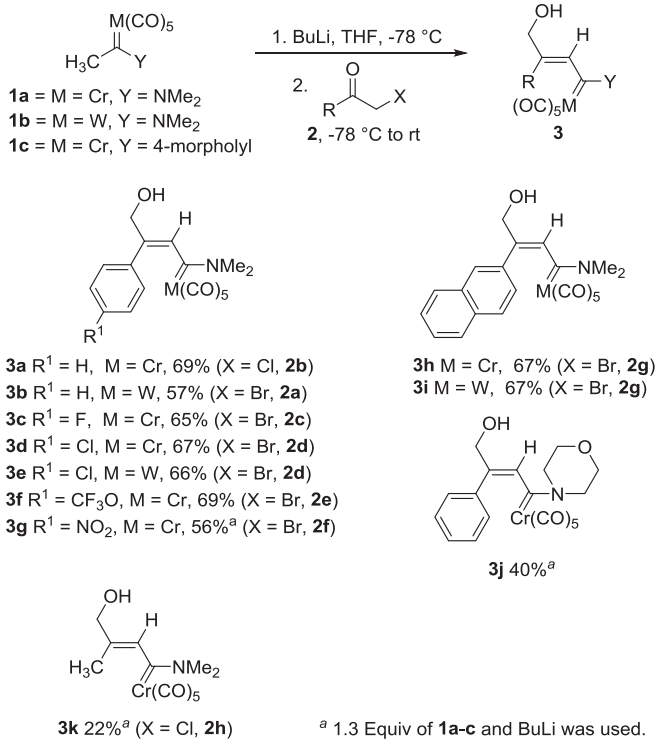
was observed in the crude reaction mixture, indicating that deprotonation of the complex **1a** did not occur under the tested conditions. The structure of the isolated alkene **3a** was determined by means of 2D NMR and nOe experiments.

The optimized reaction conditions were used to explore the scope of the developed methodology for the stereoselective synthesis of 2,2-disubstituted alkenylaminocarbene complexes **3** (Scheme 1). Thus, a 10 mol% excess of chromium and tungsten carbene complexes **1a**,**b** were treated with BuLi in dry THF followed by addition of a solution of commercially available α-haloketones in dry toluene. Phenacyl chloride (**2b**) gave a similarly high yield of the complex **3a.** The other tested substituted acetophenones **2c**–**f** gave the complexes **3c**,**d**,**f**,**g** at 56%–69% yield. It is worth noting that 1.3 equivalents of the complex **1a** and BuLi were used to prepare the alkene **3g**. The reaction conditions enabled the preparation of the tungsten complexes **3b**,**e** at yields similar to those of the chromium complexes **3a**,**d**. The synthesis of the chromium and tungsten complexes **3h**,**i** derived from 2-bromo-2′-acetonaphthone (**2g**) was also smoothly achieved. The carbene complex **1c** bearing 4-morpholyl substituent instead of *N*,*N*-dimethylamino group showed similar reactivity although 1.3 equiv of **1c** was used to obtain a 40% isolated yield for **3j**. The use of chloroacetone, as an example of aliphatic ketone, was less efficient in affording the alkene **3k** at an isolated yield of only 22%. Apart from the abovementioned α-haloketones, the reactions of the deprotonated **1a** with α-bromocyclohexanone, α-bromopropiophenone, 1-bromopinacolon or 2-bromo-2-phenylacetophenone were also tested, but no alkenes were formed under the tested conditions. Compared with the carbenes **1a**–**c**, pentacarbonyl[(*N*,*N*-dimethylamino)ethylcarbene]chromium(0) (**1d**) did not react under the tested conditions. Similarly, deprotonated *N*,*N*-dimethylacetamide and pentacarbonyl[(ethoxy)methylcarbene]chromium(0) (**1e**) did not react with phenacyl bromide under the developed conditions to afford the expected alkenes.

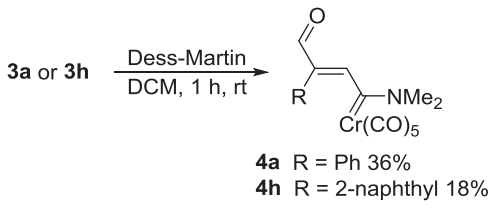
The prepared alkenes **3a**–**k** were isolated as a mixture of the desired alkenes **3** and < 10% of a compound containing aldehyde proton as observed in ^{1}H NMR spectra. When a sample from crude reaction mixture was worked up under inert atmosphere and degassed $CDCl_3$ was used, no aldehyde was present in a ^{1}H NMR spectrum. Then the aldehyde peak emerged when this sample was treated with air. Thus, we reasoned that the formed alkenes **3** were oxidized by air to the corresponding α,β-unsaturated aldehydes during workup. This assumption was supported by the oxidation of the alkenes **3a**,**h** to the aldehydes **4a** and **4h** by Dess–Martin periodinane in dichloromethane at room temperature (Scheme 2). Other tested oxidation protocols, including TEMPO oxidation [35a], Swern oxidation [35b], or PDC [35c] were unsuccessful, leading mostly to product of decomposition. We compared the ^{1}H NMR spectra of the aldehydes **4a**, **4h** with those of the alkenes **3a**,**3h**, and we found minor peaks in the isolated carbenes **3a**, **3h** that were identical to those in the aldehydes **4a**,**4h**.

We subsequently performed another set of experiments to clarify the mechanism of the observed transformation. The metalated carbene **6** reacted with an excess of the ketones **2a**,**f**, affording the epoxides **5a** and **5g** in moderate isolated yields (Scheme 3). Next, we successfully verified that **5a** can be smoothly converted into sole (*E*)-alkene **3a** by using different bases, including sodium hydroxide, potassium carbonate, DBU and metalated carbene **6**. However, the best yield for **3a** was obtained with DBU or with metalated carbene **6**.

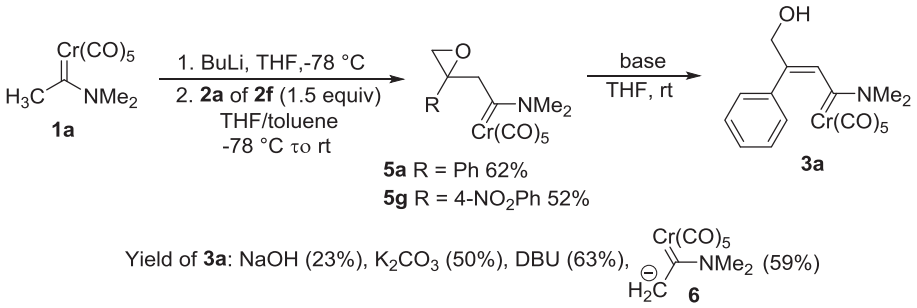
Considering the abovementioned results, we expected that the conversion of the carbenes **1** into alkenes **3** involves nucleophilic addition of **6** into α-haloketones followed by intramolecular substitution, giving epoxides **5**. According to the literature [36] on the conversion of 2,2-disubstituted epoxide into 3-hydroxyprop-2-



Scheme 1. Scope of the methodology for the synthesis of trisubstituted alkenes **3**.



Scheme 2. Oxidation of alcohols **3a**,**h** to aldehydes **4a**, **4h**.

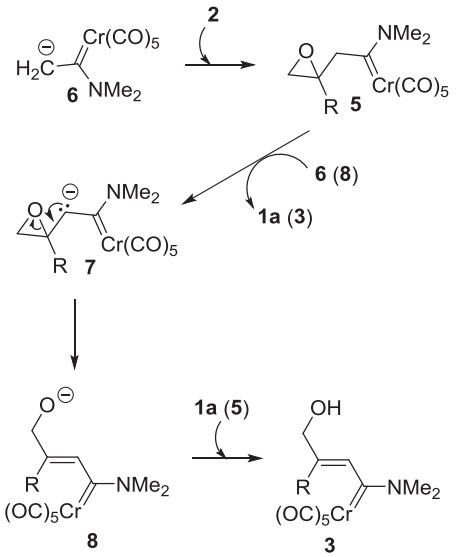


Scheme 3. Preparation of the epoxides **5a**, **5g** and conversion of **5a** into the alkene **3a**.

enoates and based on the results we obtained during the conversion of **5a** into **3a**, we presume that the epoxides **5** were deprotonated by means of the anion **6** or **8** and that the deprotonated epoxides opened to produce the trisubstituted alkene **3** (Scheme 4). However, reasons for excellent diastereoselectivity remain unclear. Detailed analysis of the crude reaction mixture between the aminocarbene complex **1a** and phenacyl bromide by ^{1}H NMR indicates the formation of alkene **3a** along with hardly identified minor by-products. This observation might indicate alternative reaction pathways including low stability of opposite (*Z*)-isomer.

3. Conclusions

In summary, we have developed a novel methodology to convert α-haloketones into trisubstituted alkenes bearing Fischer aminocarbene moiety and with good isolated yields and excellent *E*-selectivity. The reaction of the deprotonated aminocarbenes **1** in THF–toluene mixture works well with aromatic ketones, whereas aliphatic ketones are less efficient. The mechanistic proposal for this reaction consists of the formation of epoxides **5,** which rearrange into trisubstituted alkenes via a base-mediated epoxide opening process. Further mechanistic studies and synthetic application of the prepared aminocarbene complexes are currently conducted in our laboratory.



Scheme 4. Proposed mechanism for the formation of alkene **3**.

4. Experimental section

4.1. Materials and methods

All reactions were performed under an argon atmosphere. NMR spectra were measured on a Varian Gemini 300 (^{1}H, 300.07 MHz; ^{13}C, 75.46 MHz), a Bruker DRX 500 Avance (^{1}H, 500.13 MHz; ^{13}C, 125.77 MHz), or a Bruker 600 Avance^{III} (^{1}H, 600.13 MHz; ^{13}C, 150.93 MHz) spectrometer at 298 K. Mass spectra were measured on ZAB-SEQ (VG Analytical). The dry and degassed solvents were prepared by Pure-Solv MD7; silica gel (Merck, Silica Gel 60, 40–63 μm) was used for column chromatography. The carbenes **1a**–**c** were prepared by aminolysis of pentacarbonyl[(methoxy) methylcarbene]chromium(0) following the published procedure [37]. BuLi (2.5 M solution in hexane) and other compounds were purchased. Concentration of BuLi was determined by titration using menthol and 1,10-phenanthroline before use.

4.2. Synthesis of the complex *1d*

Pentacarbonyl[(*N*,*N*-dimethylamino)ethylcarbene]chromium(0) (1d). A solution of BuLi (0.87 mL, 2.2 mmol) was added to a solution of aminocarbene **1a** (0.526 g, 2.0 mmol) in dry THF (10 mL) cooled to −78 °C. The resultant mixture was stirred for 30 min at −78 °C followed by addition of methyl iodide (0.19 mL, 3.0 mmol). After 3 h at ambient temperature, the reaction mixture was diluted with ether and washed with brine (20 mL). The organic layer was dried over $MgSO_4$, concentrated under reduce pressure and column chromatography (Hexane/DCM 2/1, $R_f \approx 0.40$) afforded 0.460 g (83%) of the title compound as a yellow solid, MP 43.9–46.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.83 (s, 3H, CH_3), 3.32 (s, 3H, CH_3), 3.10 (q, $J = 8.0$ Hz, 2H, CH_2), 1.06 (t, $J = 8$ Hz, 3H, CH_3). ^{13}C NMR (126 MHz, $CDCl_3$) δ 277.9, 223.4, 217.9, 53.2, 44.9, 41.8, 9.3. IR (ATR): ν 2978 (w), 2942 (w), 2051 (m), 1965 (w), 1939 (w), 1866 (s), 1531 (m), 1398 (m), 1373 (m), 1297 (w), 1220 (w), 1160 (w), 1052 (w), 1006 (w) cm^{-1}. HR MS (ESI) $[M - H]^-$ calcd for $C_{10}H_{11}CrNO_5$: 275.9970, found 275.9969.

4.3. General procedure for the preparation of alkenes *3*

A solution of BuLi (1.1 equiv, ≈2.5 M solution in hexane) was added to a solution of aminocarbenes **1a**–**c** (1.1 equiv) in dry THF (5 mL) cooled to −78 °C. The resultant mixture was stirred for 30 min at −78 °C followed by addition of a solution of α-haloketone **2** (1.0 equiv) in dry toluene (5 mL/mmol). The reaction mixture was warmed to ambient temperature. After 3 h, the reaction mixture was diluted with ether and then washed with brine (20 mL). The organic layer was dried over $MgSO_4$, concentrated under a reduced pressure and subjected to column chromatography (silica gel) to obtain the product.

*4.3.1. (3E)-1,1,1,1,1-Pentacarbonyl-1-chroma-2-(N,N-dimethylamino)-4-phenylpenta-1,3-dien-5-ol (**3a**)*

The general procedure starting from **1a** (0.289 g, 1.1 mmol), BuLi (0.45 mL, 1.1 mmol), phenacyl bromide (0.199 g, 1.0 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/2, Rf ≈ 0.20) afforded 0.305 g (80%) of the title compound as a brown oil. 1H NMR (500 MHz, $CDCl_3$) δ 7.41–7.38 (m, 2H, CH), 7.33–7.30 (m, 1H, CH), 7.27–7.25 (m, 2H, CH), 6.96 (s, 1H, CH), 4.63–4.54 (m, 2H, CH_2), 3.70 (s, 3H, CH_3), 3.02 (s, 3H, CH_3), 1.96 (br s, 1H, OH). ^{13}C NMR (126 MHz, $CDCl_3$) δ 267.5, 223.2, 217.6, 137.2, 135.6, 128.8, 127.9, 127.2, 126.7, 65.9, 50.6, 46.0. IR (ATR): ν 3228 (w), 2050 (w), 1878 (s), 1684 (w), 1597 (w), 1537 (w), 1493 (w), 1442 (w), 1401 (w), 1223 (w), 1162 (w), 1096 (w), 1075 (w), 1049 (w), 1025 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{17}H_{15}CrNO_6$: 380.0232, found 380.0230.

*4.3.2. (3E)-1,1,1,1,1-Pentacarbonyl-2-(N,N-dimethylamino)-4-phenyl-1-tungstapenta-1,3-dien-5-ol (**3b**)*

The general procedure starting from **1b** (0.435 g, 1.1 mmol), BuLi (0.45 mL, 1.1 mmol), phenacyl bromide (0.199 g, 1.0 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/2, R_f ≈ 0.20) afforded 0.290 g (57%) of the title compound as a brown solid, MP 58.0–64.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.34 (m, 2H, CH), 7.29–7.24 (m, 3H, CH), 6.83 (s, 1H, CH), 4.66–4.53 (m, 2H, CH_2), 3.61 (s, 3H, CH_3), 2.96 (s, 3H, CH_3), 1.97 (t, J = 4.5 Hz, 1H, OH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 249.4 ($J_{183W-13C}$ = 129 Hz), 203.2 ($J_{183W-13C}$ = 128 Hz), 198.3 ($J_{183W-13C}$ = 128 Hz), 136.9, 136.6, 128.7, 128.5, 127.9, 127.3, 65.5, 52.8, 44.4. IR (ATR): ν 3212 (w), 2058 (m), 1964 (w), 1866 (s), 1537 (m), 1493 (m), 1441 (m), 1399 (m), 1318 (w), 1229 (w), 1163 (w), 1098 (w), 1076 (w), 1051 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{17}H_{15}WNO_6$: 512.0336, found 512.0339.

*4.3.3. (3E)-1,1,1,1,1-Pentacarbonyl-1-chroma-4-(4-fluorophenyl)-2-(N,N-dimethylamino)penta-1,3-dien-5-ol (**3c**)*

The general procedure starting from **1a** (0.289 g, 1.1 mmol), BuLi (0.45 mL, 1.1 mmol), 2-bromo-4′-fluoroacetophenone (0.217 g, 1.0 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/2, R_f ≈ 0.20) afforded 0.260 g (65%) of the title compound as a brown foam. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24–7.19 (m, 2H, CH), 7.08–7.02 (m, 2H, CH), 6.90 (s, 1H, CH), 4.49–4.45 (m, 2H, CH_2), 3.69 (s, 3H, CH_3), 3.03 (s, 3H, CH_3), 1.70 (t, J = 6.0 Hz, 1H, OH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 267.6, 223.0, 217.4, 162.0 (d, J = 249.1 Hz), 135.7, 133.1 (d, J = 3.80 Hz), 128.9 (d, J = 8.2 Hz), 125.4, 115.7 (d, J = 21.6 Hz), 65.9, 50.5, 45.9. IR (ATR): ν 3230 (w), 2926 (w), 2051 (w), 1972 (w), 1882 (s), 1602 (w), 1537 (w), 1508 (w), 1443 (w), 1401 (w), 1225 (w), 1160 (w), 1091 (w), 1050 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{17}H_{14}CrFNO_6$: 398.0138, found 398.0135.

*4.3.4. (3E)-1,1,1,1,1-Pentacarbonyl-4-(4-chlorophenyl)-1-chroma-2-(N,N-dimethylamino)penta-1,3-dien-5-ol (**3d**)*

The general procedure starting from **1a** (0.289 g, 1.1 mmol), BuLi (0.45 mL, 1.1 mmol), 2-bromo-4′-chloroacetophenone (0.233 g, 1.0 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/2, R_f ≈ 0.15) afforded 0.278 g (67%) of the title compound as a brown foam. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35–7.31 (m, 2H, CH), 7.20–7.15 (m, 2H, CH), 6.91 (s, 1H, CH), 4.59–4.45 (m, 2H, CH_2), 3.70 (s, 3H, CH_3), 3.03 (s, 3H, CH_3), 1.71 (t, J = 6.0 Hz, 1H, OH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 267.6, 223.0, 217.4, 136.0, 135.5, 133.7, 128.9, 128.4, 125.1, 65.7, 50.6, 46.0. IR (ATR): ν 3369 (w), 2945 (w), 2051 (w), 1973 (w), 1881 (s), 1592 (w), 1536 (w), 1491 (w), 1447 (w), 1401 (w), 1223 (w), 1162 (w), 1091 (w), 1012 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{17}H_{14}ClCrNO_6$: 413.98420, found 413.98423.

*4.3.5. (3E)-1,1,1,1,1-Pentacarbonyl-4-(4-chlorophenyl)-2-(N,N-dimethylamino)-1-tungstapenta-1,3-dien-5-ol (**3e**)*

The general procedure starting from **1b** (0.435 g, 1.1 mmol), BuLi (0.43 mL, 1.1 mmol), 2-bromo-4′-chloroacetophenone (0.233 g, 1.0 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/2, R_f ≈ 0.15) afforded 0.360 g (66%) of the title compound as a yellow foam. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35–7.33 (m, 2H, CH), 7.23–7.20 (m, 2H, CH), 6.82 (s, 1H, CH), 4.62–4.51 (m, 2H, CH_2), 3.64 (s, 3H, CH_3), 3.02 (s, 3H, CH_3), 1.93–1.76 (br m, 1H, OH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 249.3 ($J_{183W-13C}$ = 89 Hz), 203.0 ($J_{183W-13C}$ = 129 Hz), 198.1 ($J_{183W-13C}$ = 128 Hz), 137.0, 135.3, 133.7, 128.8, 128.6, 126.9, 65.3, 52.8, 44.4. IR (ATR): ν 3213 (w), 2058 (w), 1970 (w), 1915 (s), 1876 (s), 1593 (w), 1540 (m), 1489 (m), 1442 (w), 1401 (w), 1227 (w), 1164 (w), 1085 (m), 1047 (m), 1012 (m) 988 (m) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{17}H_{14}ClWNO_6$: 545.9946, found 545.9943.

*4.3.6. (3E)-1,1,1,1,1-Pentacarbonyl-1-chroma-4-[4-(trifluoromethyl) phenyl]-2-(N,N-dimethylamino)penta-1,3-dien-5-ol (**3f**)*

The general procedure starting from **1a** (0.289 g, 1.1 mmol), BuLi (0.45 mL, 1.1 mmol), 4-(trifluoromethoxy)phenacyl bromide (0.283 g, 1.0 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/2, R_f ≈ 0.20) afforded 0.330 g (69%) of the title compound as a brown oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29–7.19 (m, 4H, CH), 6.93 (s, 1H, CH), 4.57–4.52 (m, 2H, CH_2), 3.71 (s, 3H, CH_3), 3.05 (s, 3H, CH_3), 1.73 (t, J = 6.0 Hz, 1H, OH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 268.0, 222.9, 217.3, 148.5 (q, J = 2.0 Hz), 136.2, 135.7, 128.6, 124.8, 121.0, 124.2–119.1 (q, J = 257.3 Hz) 65.8, 50.5, 46.0. IR (ATR): ν 3369 (w), 2053 (w), 1973 (w), 1881 (s), 1685 (w), 1602 (w), 1539 (w), 1508 (w), 1450 (w), 1403 (w), 1251 (w), 1209 (w), 1156 (w), 1017 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{18}H_{14}CrF_3NO_7$: 464.0055, found 464.0052.

*4.3.7. (3E)-1,1,1,1,1-Pentacarbonyl-1-chroma-2-(N,N-dimethylamino)-4-(4-nitrophenyl)penta-1,3-dien-5-ol (**3g**)*

The general procedure starting from **1a** (0.342 g, 1.3 mmol), BuLi (0.51 mL, 1.3 mmol), 2-bromo-4′-nitroacetophenone (0.244 g, 1.0 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/1, R_f ≈ 0.35) afforded 0.240 g (56%) of the title compound as a red foam. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.23–8.20 (m, 2H, CH), 7.44–7.41 (m, 2H, CH), 7.03 (s, 1H, CH), 4.67–4.53 (m, 2H, CH_2), 3.76 (s, 3H, CH_3), 3.11 (s, 3H, CH_3), 1.79 (t, J = 6.0 Hz, 1H, OH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 268.2, 222.6, 217.1, 146.7, 143.8, 137.7, 127.9, 123.8, 123.4, 65.5, 50.6, 46.2. IR (ATR): ν 3606 (m), 2943 (m), 2051 (m), 1894 (s), 1694 (m), 1592 (m), 1506 (m), 1399 (w), 1332 (m), 1106 (w), 1006 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{17}H_{14}CrN_2O_8$: 425.0083, found 425.0085.

*4.3.8. (3E)-1,1,1,1,1-Pentacarbonyl-1-chroma-2-(N,N-dimethylamino)-4-(2-naphthyl)penta-1,3-dien-5-ol (**3h**)*

The general procedure starting from **1a** (0.579 g, 2.2 mmol), BuLi (0.87 mL, 1.1 mmol), 2-bromo-2′-acetonaphthone (0.498 g, 2.0 mmol), THF (10 mL), toluene (10 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/2, R_f ≈ 0.15) afforded 0.498 g (67%) of the title compound as a brown foam. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84–7.79 (m, 4H, CH), 7.71 (s, 1H, CH), 7.52–7.47 (m, 2H, CH), 7.38–7.35 (m, 1H, CH), 7.02 (s, 1H, CH), 4.73–4.59 (m, 2H, CH_2), 3.65 (s, 3H, CH_3), 2.94 (m, 3H, CH_3), 1.75 (t, J = 6.0 Hz, 1H, OH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 266.6, 223.1, 217.5, 135.7, 134.4, 133.2, 132.4, 128.4, 128.1, 127.5, 126.55, 126.53, 126.49, 126.3, 124.4, 65.9, 50.5, 45.8. IR (ATR): ν 3323 (m), 2050 (m), 1880 (s), 1535 (m), 1504 (w), 1439 (w), 1399 (w), 1222 (w), 1159 (w), 1130 (w), 1080 (w), 1047 (w), 1010 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{21}H_{17}CrNO_6$: 430.0388, found 430.0387.

*4.3.9. (3E)-1,1,1,1,1-Pentacarbonyl-2-(N,N-dimethylamino)-4-(2-naphthyl)-1-tungstapenta-1,3-dien-5-ol (**3i**)*

The general procedure starting from **1b** (0.435 g, 1.1 mmol), BuLi (0.43 mL, 1.1 mmol), 2-bromo-2′-acetonaphthone (0.249 g, 1.0 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/2, $R_f \approx 0.15$) afforded 0.380 g (67%) of the title compound as a yellow foam. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85–7.81 (m, 3H, CH), 7.76–7.75 (m, 1H, CH), 7.52–7.48 (m, 2H, CH), 7.42–7.39 (m, 1H, CH), 6.94 (m, 1H, CH), 4.77–4.64 (m, 2H, CH_2), 3.58 (s, 3H, CH_3), 2.91 (s, 3H, CH_3), 1.96 (t, $J = 6.0$ Hz, 1H, CH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 249.0 ($J_{183W\text{-}13C} = 88$ Hz), 203.1 ($J_{183W\text{-}13C} = 128$ Hz), 198.4 ($J_{183W\text{-}13C} = 128$ Hz), 136.9, 134.3, 133.2, 132.5, 128.4, 128.2, 128.1, 127.5, 126.8, 126.7, 126.6, 124.6, 65.7, 52.8, 44.4. IR (ATR): ν 3181 (w), 2057 (m), 1876 (s), 1540 (m), 1441 (w), 1400 (w), 1227 (w), 1163 (w), 1091 (w), 1052 (w), 1002 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{21}H_{17}WF_3NO_6$: 562.0492, found 562.0497.

*4.3.10. (3E)-1,1,1,1,1-Pentacarbonyl-1-chroma-2-(4-morpholyl)-4-phenylpenta-1,3-dien-5-ol (**3j**)*

The general procedure starting from **1c** (0.397 g, 1.3 mmol), BuLi (0.51 mL, 1.3 mmol), phenacyl bromide (0.199 g, 1.0 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/1, $R_f \approx 0.40$) afforded 0.170 g (40%) of the title compound as an orange foam. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40–7.23 (m, 5H, CH), 6.96 (s, 1H, CH), 4.62–4.49 (m, 2H, CH_2), 4.24–4.21 (m, 2H, CH_2), 3.93–3.86 (m, 1H, CH_2), 3.66–3.48 (m, 3H, CH_2), 3.40–3.33 (m, 1H, CH_2), 2.47–2.42 (m, 1H, CH_2), 1.76 (t, $J = 6.0$ Hz, 1H, OH). ^{13}C NMR (101 MHz, $CDCl_3$) δ 266.5, 223.0, 217.4, 136.8, 134.1, 128.8, 128.2, 127.5, 126.4, 66.6, 65.8, 65.6, 59.9, 54.6. IR (ATR): ν 3403 (w), 2925 (w), 2859 (w), 2051 (m), 1973 (w), 1879 (s), 1685 (w), 1508 (w), 1441 (w), 1302 (w), 1261 (w), 1245 (w), 1109 (w), 1056 (w), 1021 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{19}H_{17}CrNO_7$: 422.0337, found 422.0338.

*4.3.11. (3E)-1,1,1,1,1-Pentacarbonyl-1-chroma-2-(N,N-dimethylamino)-4-methylpenta-1,3-dien-5-ol (**3k**)*

The general procedure starting from **1a** (0.342 g, 1.3 mmol), BuLi (0.51 mL, 1.3 mmol), chloroacetone (0.092 g, 1.0 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 1/1 → EtOAc/hexane 1/1 → 2/1, $R_f \approx 0.20$) afforded 0.070 g (22%) of the title compound as a brown oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.55 (s, 1H, CH), 4.13 (br m, 2H, CH_2), 3.88 (s, 3H, CH_3), 3.36 (s, 3H, CH_3), 1.45 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 271.3, 223.5, 217.5, 135.2, 123.1, 66.6, 50.6, 45.0, 14.6. IR (ATR): ν 3388 (w), 2920 (w), 2052 (w), 1878 (s), 1681 (w), 1607 (w), 1537 (w), 1444 (w), 1402 (w), 1224 (w), 1162 (w), 1020 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{12}H_{13}CrNO_6$: 318.0075, found 318.0073.

*4.4. Synthesis of aldehydes **4a** and **4h***

*4.4.1. (3E)-1,1,1,1,1-Pentacarbonyl-1-chroma-2-(N,N-dimethylamino)-4-phenylpenta-1,3-dien-5-al (**4a**)*

Dess-Martin periodinane (0.192 g, 0.45 mmol) was added to a solution of the carbene **3a** (0.157 g, 0.41 mmol) in dry DCM (6 mL). The resultant mixture was stirred for 1 h at ambient temperature. Then 1 mL of isopropyl alcohol was added and the reaction mixture was stirred 30 min at ambient temperature, concentrated under reduce pressure and column chromatography (Hexane/DCM 1/2 → DCM, $R_f \approx 0.70$) afforded 0.056 g (36%) of the title compound as a red oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.72 (s, 1H, CH), 7.41–7.33 (m, 4H, CH), 7.25–7.22 (m, 2H, CH), 3.75 (s, 3H, CH_3), 3.08 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 264.5, 222.5, 216.5, 191.5, 149.9, 131.8, 128.5, 128.4, 128.3, 125.6, 50.2, 47.6. IR (ATR): ν 2836 (w), 2716 (w), 2053 (m), 1889 (s), 1687 (s), 1589 (m), 1575 (m) 1556 (m), 1493 (m), 1443 (m), 1403 (m), 1361 (m), 1225 (w), 1203 (w), 1165 (w), 1088 (w), 1073 (w), 1017 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{17}H_{13}CrNO_6$: 378.0075, found 378.0072.

*4.4.2. (3E)-1,1,1,1,1-Pentacarbonyl-1-chroma-2-(N,N-dimethylamino)-4-(2-naphthyl)penta-1,3-dien-5-al (**4h**)*

Dess-Martin periodinane (0.622 g, 1.47 mmol) was added to a solution of the carbene **3h** (0.575 g, 1.33 mmol) in dry DCM (6 mL). The resultant mixture was stirred for 1 h at ambient temperature. Then 1 mL of isopropyl alcohol was added and the reaction mixture was stirred 30 min at ambient temperature, concentrated under reduce pressure and column chromatography (Hexane/DCM 1/2 → DCM, $R_f \approx 0.80$) afforded 0.100 g (18%) of the title compound as a red foam. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.76 (s, 1H, CH), 7.86–7.82 (m, 3H, CH), 7.75 (s, 1H, CH), 7.54–7.48 (m, 3H, CH), 7.38–7.35 (m, 1H, CH), 3.66 (s, 3H, CH_3), 2.94 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 263.2, 222.5, 216.5, 191.6, 150.0, 132.9, 132.6, 129.2, 128.1, 127.98, 127.96, 127.5, 126.8, 126.4, 125.4, 125.0, 50.1, 47.4. IR (ATR): ν 3059 (w), 2053 (m), 1978 (w), 1882 (s), 1678 (m), 1543 (m), 1502 (w), 1440 (w), 1403 (w), 1222 (w), 1159 (w), 1132 (w), 1071 (w), 1018 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{21}H_{15}CrNO_6$: 428.0232, found 428.0230.

*4.5. Preparation of epoxides **5a** and **5g***

*4.5.1. 1,1,1,1,1-Pentacarbonyl-1-chroma-4,5-epoxy-2-(N,N-dimethylamino)-4-phenylpent-1-en (**5a**)*

The general procedure starting from **1a** (0.263 g, 1.0 mmol), BuLi (0.39 mL, 1.0 mmol), phenacyl bromide (0.299 g, 1.5 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/EtOAc 19/1 → DCM/hexane 1/2, $R_f \approx 0.35$) gave 0.236 g (62%) of the title compound as a red solid, MP > 115.6 °C (decomp). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48–7.46 (m, 2H, CH), 7.41–7.34 (m, 3H, CH), 4.37 (d, $J = 10.5$ Hz, 1H, CH_2), 3.88 (s, 3H, CH_3), 3.58 (d, $J = 10.5$ Hz, 1H, CH_2), 3.44 (s, 3H, CH_3), 3.06 (d, $J = 3.6$ Hz, 1H, CH_2), 2.81 (d, $J = 3.6$ Hz, 1H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 269.2, 222.7, 217.5, 140.3, 128.6, 128.1, 125.5, 58.8, 53.7, 53.5, 53.0, 44.8. IR (ATR): ν 3066 (w), 2051 (m), 1962 (w), 1872 (s), 1543 (m), 1495 (w), 1474 (m), 1448 (w), 1399 (m), 1362 (w), 1291 (w), 1229 (w), 1138 (w), 1063 (w), 1035 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{17}H_{15}CrNO_6$: 380.0232, found 380.0230.

*4.5.2. 1,1,1,1,1-Pentacarbonyl-1-chroma-4,5-epoxy-2-(N,N-dimethylamino)-4-(4-nitrophenyl)pent-1-en (**5g**)*

The general procedure starting from **1a** (0.263 g, 1.0 mmol), BuLi (0.39 mL, 1.0 mmol), 2-bromo-4′-nitroacetophenone (0.366 g, 1.5 mmol), THF (5 mL), toluene (5 mL), column chromatography (Hexane/DCM 2/1 → 1/2, $R_f \approx 0.25$) gave 0.220 g (52%) of the title compound as a brown solid, MP > 93.0 °C (decomp). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.27–8.24 (m, 2H, CH_2), 7.67–7.64 (m, 2H, CH), 4.41 (d, $J = 14.7$ Hz, 1H, CH_2), 3.90 (s, 3H, CH_3), 3.57 (d, $J = 14.1$ Hz, 1H, CH_2), 3.45 (s, 3H, CH_3), 3.13 (d, $J = 4.5$ Hz, 1H, CH_2), 2.79 (d, $J = 4.5$ Hz, 1H, CH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 268.2, 222.4, 217.4, 147.6, 147.2, 126.6, 123.9, 58.3, 53.8, 53.2, 52.4, 44.9. IR (ATR): ν 2920 (w), 2052 (m), 1903 (s), 1875 (s), 1635 (w), 1599 (m), 1542 (m), 1512 (m), 1402 (m), 1345 (m), 1302 (w), 1286 (w), 1228 (w), 1145 (w), 1112 (w), 1053 (w), 1013 (w) cm^{-1}. HR MS (ESI) $[M-H]^-$ calcd for $C_{17}H_{14}CrN_2O_8$: 425.0083, found 425.0082.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

Financial support from specific university research (MŠMT No 21-SVV/2019) and the Grant Agency of the Czech Republic (grant no. 17-21770S) is acknowledged.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jorganchem.2019.120971.

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