

Solid-Support-Bound 1-Aminoimidazolium Chlorochromate: A Selective, Efficient and Recyclable Oxidant

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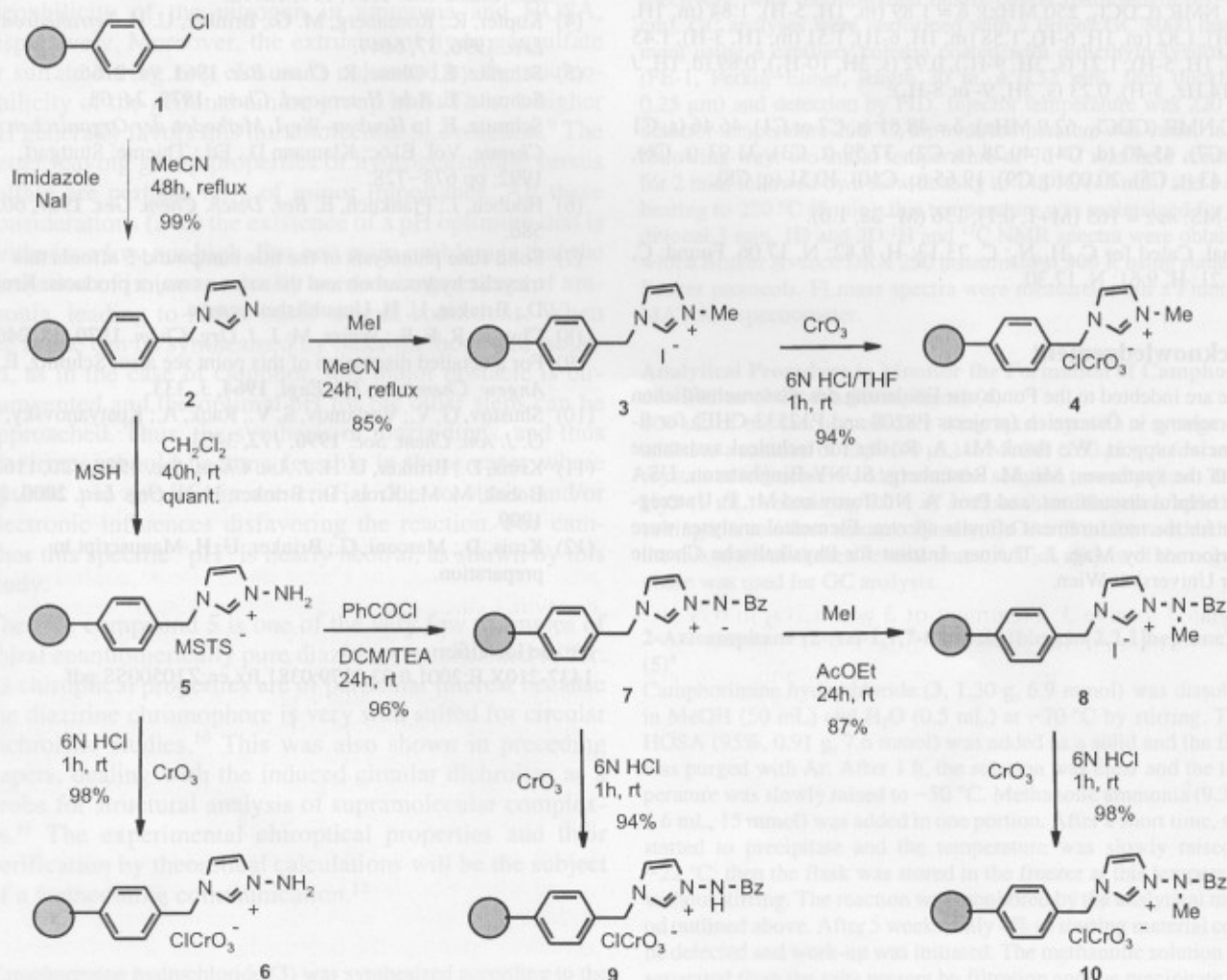
Received 23 May 2000; revised 5 October 2000

Abstract: A series of polymer-bound imidazolium chlorochromates have been synthesised and used as selective oxidants for benzylic and cinnamyl alcohols. Solid-support-bound 1-aminoimidazolium chlorochromate proved to be the most convenient system due to its efficiency, selectivity and ease of recycling the material.

Key words: alcohols, chromium, oxidations, polymers

The use of polymer-bound reagents in solution phase chemistry allows reactions to be driven to completion and product isolation by filtration. Three types of polymeric

reagents have been distinguished:^{1–3} (a) Polymeric supports carrying a functional group to be transferred to the ligand; (b) Polymer-bound reagents that catalyse a conversion; and (c) Polymer-bound reagents that remove an excess of reagent or product from the reaction mixture (scavengers or resin capture). Within the first group, several oxidants have been reported including chromate,^{4–7} chlorochromate,⁸ dichromate,⁹ osmium tetroxide,¹⁰ periodate,^{11,12} perruthenate,^{13,14} and peracid.¹⁵ Some of these compounds have been used to perform oxidations of primary and secondary alcohols. The reagents have been supported on organic polymers such as polyvinylpyri-



Scheme 1

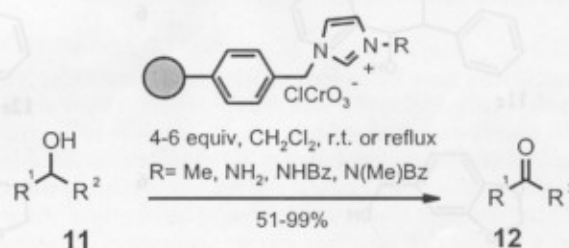
dine,^{8,9} Merrifield's resin⁵ or Amberlyst ion exchange resin^{6,7,13} and these systems were not selective with respect to the oxidation of allylic or benzylic alcohols in any case. Poly(vinylpyridinium chlorochromate)⁸ (PVPCC) and poly(vinylpyridinium dichromate)⁹ (PVPDC) proved to be efficient and recyclable oxidants for alcohols, although they are not selective. Other chlorochromate and dichromate reagents have been supported by adsorption onto inorganic materials such as alumina¹⁶ and molecular sieves¹⁷ and they were also shown to be efficient, non-selective oxidants. PVPDC has recently been used together with other polymeric reagents in a simultaneous multistep synthesis.¹⁸ Polymer-bound oxidants based on polyvinylpyridine *N*-oxide dichromate,¹⁹ polyvinylpyridine silver dichromates²⁰ and a pyrazine-based polymeric complex of oxodiperoxochromium(VI)²¹ have also been described. Also recently, other immobilised oxidants such as hypervalent iodine^{22–25} and TEMPO^{26–29} systems have been reported. Specially, polymer-supported (diacetoxyiodo)benzene (PSDIB) is able to oxidise a variety of organic compounds with good to quantitative yield in both organic^{22,23} and aqueous solvents.²⁵ PSDIB has been also used in a multi-step sequence of polymer supported reagents for the synthesis of alkaloids.²⁴ To our knowledge, although there is a set of polymer-supported oxidants currently available, the solid phase selective oxidation of allylic and benzylic alcohols is not reported so far.

We recently reported the synthesis of an imidazolium-based chlorochromate [1-(benzoylamino)-3-methylimidazolium chlorochromate: BAMICC], which was shown to be a selective oxidant for benzylic and allylic alcohols.³⁰ A number of specific features of this system, together with the advantages of polymer-bound reagents in solution phase chemistry, prompted us to explore the application of solid-support-bound imidazolium chlorochromate derivatives for the selective oxidation of alcohols. We report here the synthesis of a series of polymer-bound chlorochromates, their application to the oxidation of alcohols and, for the most efficient example, the recycling procedure.

The preparation of our polymer-bound chlorochromates (Scheme 1) was carried out starting from Merrifield's resin **1** (2% crosslinking). The reaction with imidazole in the presence of NaI quantitatively gave **2**, which was then methylated to give **3**. Treatment of **3** with CrO₃ in 6 N HCl yielded the chlorochromate **4** in high yield. The preparation of chlorochromate resins based on *N*-aminoimidazolium was carried out by amination of **2** with *O*-(mesitylsulfonyl)hydroxylamine (MSH; **Caution!** explosions have been reported^{31,32} involving this reagent and so it is strongly recommended that MSH be prepared immediately prior to use and that it should not be stored) to give **5**, and further chlorochromate formation to give **6**. Derivatives of **6** were prepared by benzoylation of **5**, which gave the ylide **7**, followed by further methylation to give **8**. Chlorochromate formation from **7** and **8** yielded **9** and **10**, respectively. Yields and conversions for com-

pounds **2–10** were determined based on weight gain, Volhard titration³³ and microanalysis.³⁴

We first screened the reactions of **4**, **6**, **9** and **10** with different alcohols (aliphatic, allylic, benzylic) to study the scope of the oxidation (Scheme 2, Table 1). We used standard conditions (CH₂Cl₂, reflux, 24 h, 4 equiv of oxidant) to compare the oxidative ability of the resins. We found that these reagents do not oxidise aliphatic or non-conjugated allylic alcohols, however, they can selectively oxidise benzylic and cinnamyl alcohols. After testing the oxidants with **11a** (Table 1, Entries 1–4), resins **4** and **6** proved to be more efficient than **9** and **10**. Although the reaction mixture was refluxed for 24 hours, in the case of resin **6** the reaction was complete in 7 hours. Resins **4** and **6** were then tested with alcohols **11b** (Table 1, Entries 5 and 6), **11c** (Table 1, Entries 7 and 8), **11d** (Table 1, Entries 9 and 10) and **11e** (Table 1, Entries 11 and 12), with **6** being the best oxidant tested so far. Moreover, in the oxidation of **11e** with **4** to give **12e**, a significant further oxidation to give benzil was observed, while the same reaction performed with **6** cleanly yielded **12e**.



Scheme 2

Other benzylic and cinnamyl alcohols were oxidised with **6** (Table 1, Entries 13–21) and gave high yields of the corresponding aldehydes and ketones. One exception was **11g**, which yielded **12g** in only moderate yield (Table 1, Entry 14). A disappointing result was obtained in the oxidation of **11i**,^{35,36} which led to complete *cis-trans* isomerisation to **12i** (Table 1, Entry 19). Finally, diol **11n**, bearing both aliphatic and benzylic alcohol groups, behaved as expected and was selectively oxidised only in the benzylic hydroxy group to give **12n**, which has previously been prepared through a Williamson reaction between *p*-hydroxybenzaldehyde and 2-chloropropan-1-ol.³⁷ Reactions were monitored by TLC and were continued until the starting material had been completely consumed. Workup simply involved filtering and washing the resin and evaporating the filtrate to dryness. All the products apart from **12g**, which needed to be chromatographed, gave clean ¹H NMR spectra that exactly matched those of authentic samples (with the exception of **12n**, all products are commercially available from Aldrich Co.). These oxidations can be also performed at room temperature (Table 1, Entries 2 and 16), but longer reaction times and a larger excess of **6** are required to obtain comparable yields.

Table 1 Oxidation of Alcohols by Solid-Support-Bound Chlorochromates

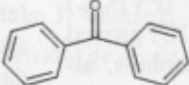
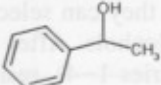
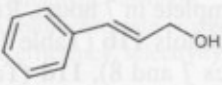
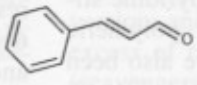
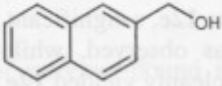
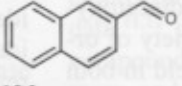
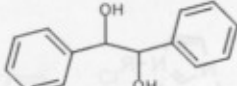
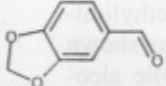
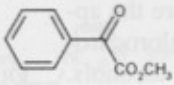
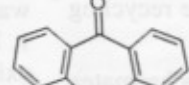
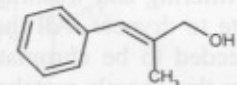
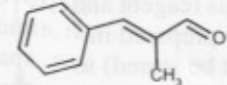
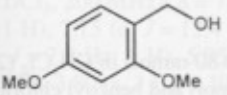
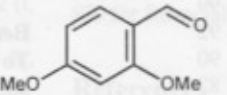
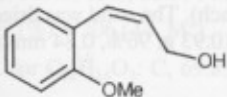
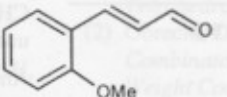
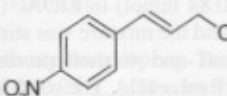
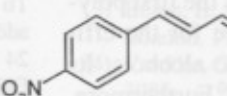
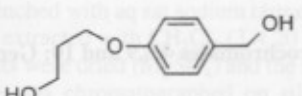
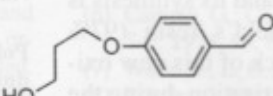
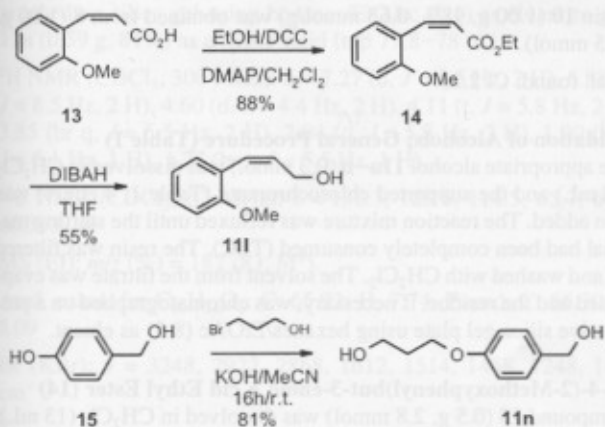
Entry	Alcohol 11	Oxidant	Carbonyl Compound 12	Time reflux/r.t. (h)	Yield (%) ^a
1	 11a	4	 12a	24	85
2		6		24 ^b /33 ^c	97 ^d
3		9		24	56
4		10		24	51
5	 11b	4	 12b	24	51
6		6		24	82 ^e
7	 11c	4	 12c	24	63
8		6		24	91 ^f
9	 11d	4	 12d	24	75
10		6		24	96 ^g
11	 11e	4	 12e	24	63 ^h
12		6		24	96
13	 11f	6	 12f	2.2	88 ⁱ
14	 11g	6	 12g	6	54
15	 11h	6	 12h	2.5	95
16	 11i	6	 12i	3/33 ^c	99
17	 11j	6	 12j	3.5	94

Table 1 (continued)

Entry	Alcohol 11	Oxidant	Carbonyl Compound 12	Time reflux/r.t. (h)	Yield (%) ^a
18		6		6.3	97
19		6		4.5	84
20		6		2.8	95
21		6		1.8	78 ⁱ

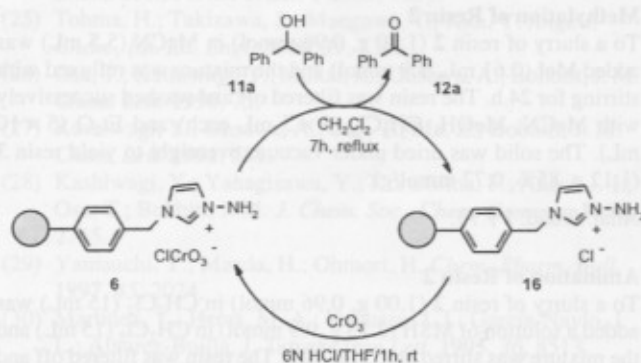
^a Yield of isolated product.^b This reaction was complete after 7 h reflux.^c 6 equiv of **6** were added for completion at r.t.^d Lit.^{17,21} 100%.^e Lit.^{8,21} 96%, Lit.^{9,25} quant., Lit.¹³ 60%, Lit.¹⁸ 55%.^f Lit.⁸ 100%, Lit.^{9,16} 98%, Lit.¹³ 95%, Lit.²¹ 90%.^g Lit.²¹ 99%.^h Benzil (37%) was also obtained.ⁱ Lit.^{13,21} 95%.^j Lit.³⁷ oil.

Compound **11l**^{35,36} was synthesised, from commercially available **13**, through esterification to give **14**³⁷ followed by further reduction. Compound **11n** was synthesised by a Williamson reaction between *p*-hydroxybenzyl alcohol (**15**) and 3-bromopropan-1-ol (Scheme 3).



Scheme 3

Resin **6** was regenerated and recycled six times in the reaction with **11a** (Scheme 4). After each process, the solid-support-bound 1-aminoimidazolium chloride **16** was regenerated by treatment with CrO_3/HCl and then recycled. Each reaction cycle led to efficient recovery of **6** and the product **12a** was obtained in high yields, as summarised in Table 2. Higher yields for cycles 2–6 can be obtained by employing longer reaction times. Moreover, **6** showed to be stable at least after one year at -12°C or eight months at room temperature when stored in the dark.



Scheme 4

Table 2 Recycling of polymer-bound chlorochromate **6**

Cycle	6 (mg)/Recovery (%) ^a	11a (mmol)	Yield (%) ^b
1	540/95	0.212	99
2	530/93	0.200	92
3	520/91	0.196	90
4	490/86	0.195	82
5	470/82	0.187	79
6	460/81	0.183	73

^a Ratio **6**/Alcohol = 4:1. Initial amount of **6** was 570 mg.^b Yield for isolated product.

In conclusion, we have shown that resin **6** is the first polymer-bound chlorochromate that is selective for the efficient oxidation of benzylic and cinnamyl alcohols. Its selectivity is greater than that of BAMICC.³⁰ Furthermore, we have shown that this material can be efficiently regenerated and recycled, it is stable enough and its synthesis is straightforward starting from Merrifield's resin (97% overall yield, three steps). One drawback of this new oxidant is the concomitant *cis-trans* isomerisation during the oxidation of (*Z*)-cinnamyl alcohols, which has been reported to occur for BAMICC with allylic alcohols.³⁰

NMR spectra were recorded on Varian Gemini 250 and Unity 300 spectrometers using CHCl₃ (δ = 7.26 and 77.0 ppm) as internal reference. IR spectra were obtained with a Perkin-Elmer 1310 spectrophotometer. MS was performed on a Hewlett-Packard 5988 A spectrometer. Combustion elemental analysis was performed with a Perkin-Elmer analyser Model 2400. Melting points were determined on a Gallenkamp apparatus and are uncorrected. Reagents were purchased from Aldrich Co. and used as received. Flash chromatography was carried out on silica gel 60 (400–630 mesh). Preparative plate chromatography was performed on silica gel 60 plates (20 × 20 × 2 mm).

Polymer-Bound Imidazole **2**

To a slurry of resin **1** (2% crosslinking, 1.00 g, 1 mmol) in MeCN (6.5 mL) were added imidazole (0.68 g, 10 mmol) and NaI (0.01 g, 0.07 mmol) and the mixture was refluxed with stirring for 48 h. The resin was filtered off and washed successively with MeCN (5 × 5 mL), 10% aq K₂CO₃/MeOH (1:1), H₂O/MeOH (1:1), MeOH and Et₂O (5 × 10 mL each). The solid was dried under vacuum overnight to yield resin **2** (1.03 g, 99%, 0.96 mmol/g).

Anal. found: Cl 0.03, N 2.70.

Methylation of Resin **2**

To a slurry of resin **2** (1.00 g, 0.96 mmol) in MeCN (5.5 mL) was added MeI (0.61 mL, 9.9 mmol) and the mixture was refluxed with stirring for 24 h. The resin was filtered off and washed successively with MeCN, MeOH, CH₂Cl₂ (5 × 5 mL each) and Et₂O (5 × 10 mL). The solid was dried under vacuum overnight to yield resin **3** (1.12 g, 85%, 0.72 mmol/g).

Anal. found: I 9.13.

Amination of Resin **2**

To a slurry of resin **2** (1.00 g, 0.96 mmol) in CH₂Cl₂ (15 mL) was added a solution of MSH (2.13 g, 9.9 mmol) in CH₂Cl₂ (15 mL) and the mixture was stirred at r.t. for 40 h. The resin was filtered off and washed successively with CH₂Cl₂, MeOH, CH₂Cl₂ (5 × 5 mL each)

and Et₂O (5 × 10 mL). The solid was dried under vacuum overnight to yield resin **5** (1.21 g, ~100%, 0.80 mmol/g).

Anal. found: S 2.56.

Benzoylation of Resin **5**

To a slurry of resin **5** (1.00 g, 0.80 mmol) in CH₂Cl₂ (20 mL) were added TEA (1.51 mL, 10.89 mmol) and benzoyl chloride (1.15 mL, 9.9 mmol) and the mixture was refluxed with stirring for 24 h. The resin was filtered off and washed successively with CH₂Cl₂, MeOH, CH₂Cl₂ and Et₂O (3 × 10 mL each). The solid was dried under vacuum overnight to yield resin **7** (0.93 g, 96%, 0.84 mmol/g).

Anal. found: S 0.10.

Methylation of Resin **7**

To a slurry of resin **7** (1.00 g, 0.84 mmol) in EtOAc (20 mL) was added MeI (0.6 mL, 9.5 mmol) and the mixture was stirred at r.t. for 24 h. The resin was filtered off and washed successively with EtOAc, CH₂Cl₂ and Et₂O (3 × 10 mL each). The solid was dried under vacuum overnight to yield resin **8** (1.10 g, 87%, 0.65 mmol/g).

Anal. found: I 8.24.

Polymer-Bound Chlorochromates **4, 6, 9** and **10**; General Procedure

To a slurry of the appropriate resin **3**, **5**, **7** and **8**, respectively, in THF (12 mL/mmol) was added dropwise a 1 M solution of CrO₃ (10 equiv) in 6 N HCl at 0 °C. After the addition was complete the mixture was stirred at r.t. for 1 h. The resin was then filtered off and washed successively with H₂O/THF (1:1), THF, CH₂Cl₂ (5 × 6 mL/mmol each) and Et₂O (5 × 12 mL/mmol) and dried under vacuum overnight.

4

Resin **4** (1.01 g, 94%, 0.67 mmol/g) was obtained from **3** (1.00 g, 0.72 mmol).

Anal. found: Cl 2.37.

6

Resin **6** (0.94 g, 98%, 0.99 mmol/g) was obtained from **5** (1.00 g, 0.96 mmol).

Anal. found: Cl 3.50.

9

Resin **9** (1.11 g, 94%, 0.71 mmol/g) was obtained from **7** (1.00 g, 0.84 mmol).

Anal. found: Cl 2.52.

10

Resin **10** (1.00 g, 98%, 0.63 mmol/g) was obtained from **8** (1.00 g, 0.65 mmol).

Anal. found: Cl 2.23.

Oxidation of Alcohols; General Procedure (Table 1)

The appropriate alcohol **11a–n** (0.3 mmol) was dissolved in CH₂Cl₂ (20 mL) and the supported chlorochromate (Table 1, 4 equiv) was then added. The reaction mixture was refluxed until the starting material had been completely consumed (TLC). The resin was filtered off and washed with CH₂Cl₂. The solvent from the filtrate was evaporated and the residue, if necessary, was chromatographed on a preparative silica gel plate using hexanes/EtOAc (8:2) as eluent.

cis-4-(2-Methoxyphenyl)but-3-enoic Acid Ethyl Ester (**14**)

Compound **13** (0.5 g, 2.8 mmol) was dissolved in CH₂Cl₂ (13 mL), and EtOH (0.17 mL, 3 mmol) and DMAP (34 mg, 0.28 mmol) were added. The mixture was cooled to 0 °C and DCC (0.63 g, 3.08 mmol) was added. The mixture was stirred for 19 h at r.t., Et₂O (50 mL) was added, the solids were filtered off and the filtrate was

evaporated to dryness. The residue was chromatographed on silica gel using hexanes/EtOAc (9:1) as eluent. Compound **14** (0.51 g, 88%) was obtained as a yellowish oil.

¹H NMR (CDCl₃, 200 MHz): δ = 7.53 (d, *J* = 7.8 Hz, 1 H), 7.32 (t, *J* = 7.8 Hz, 1 H), 7.15 (d, *J* = 12.4 Hz, 1 H), 6.93 (t, *J* = 7.8 Hz, 1 H), 6.85 (d, *J* = 7.8 Hz, 1 H), 5.95 (d, *J* = 12.4 Hz, 1 H), 4.15 (q, *J* = 6.9 Hz, 2 H), 3.80 (s, 3 H), 1.30 (t, *J* = 6.9 Hz, 3 H).

¹³C NMR (CDCl₃, 62 MHz): δ = 166.3, 157.0, 138.9, 130.6, 130.2, 124.0, 119.9, 119.8, 60.0, 55.3, 14.0.

MS (EI): *m/z* (%) = 206 (44, M⁺).

Anal. calcd. for C₁₂H₁₄O₃: C, 69.89; H, 6.84. Found: C, 69.54; H, 7.23.

IR (CHCl₃): ν = 2937, 2840, 1720, 1629, 1599, 1577, 1487, 1437, 1185, 1159, 1110 cm⁻¹.

cis-3-(2-Methoxyphenyl)prop-2-en-1-ol (**11l**)

Compound **14** (0.25 g, 1.21 mmol) was dissolved in THF (4 mL) under argon, cooled to 0 °C and 1 M DIBAL in hexane (2.42 mL, 2.42 mmol) was added. The mixture was stirred at r.t. for 20 h, the reaction quenched with aq sat sodium tartrate solution (50 mL) and the mixture extracted with CH₂Cl₂ (3 × 20 mL). The combined organic extracts were dried (MgSO₄) and the solvent was evaporated. The residue was chromatographed on silica gel using hexanes/EtOAc (9:1 to 7:3) to give **11l** (0.11 g, 55%) as a colourless oil.

¹H NMR (CDCl₃, 300 MHz): δ = 7.26 (dt, *J* = 7.7 Hz, *J* = 1.6 Hz, 1 H), 7.10 (dd, *J* = 7.7 Hz, *J* = 1.6 Hz, 1 H), 6.93 (t, *J* = 7.7 Hz, 1 H), 6.88 (d, *J* = 7.7 Hz, 1 H), 6.68 (d, *J* = 11.3 Hz, 1 H), 5.92 (dt, *J* = 11.3 Hz, *J* = 6.8 Hz, 1 H), 4.31 (dd, *J* = 6.8 Hz, *J* = 1.2 Hz, 2 H), 3.83 (s, 3 H), 1.95 (s, 1 H).

¹³C NMR (CDCl₃, 75 MHz): δ = 156.8, 130.9, 130.2, 128.7, 126.7, 125.3, 120.1, 110.4, 59.8, 55.4.

MS (EI): *m/z* (%) = 164 (59, M⁺).

Anal. calcd. for C₁₀H₁₂O₂: C, 73.15; H, 7.37. Found: C, 72.83; H, 7.55.

IR (CHCl₃): ν = 3340, 2934, 1597, 1487, 1461, 1435, 1245, 1109, 1022, 754 cm⁻¹.

3-(4-Hydroxymethylphenoxy)propan-1-ol (**11n**)

To a solution of **15** (0.5 g, 4 mmol) in MeCN (2.5 mL) was added powdered KOH (0.25 g, 4.4 mmol) and the mixture was stirred for 30 min at r.t. 3-Bromopropanol (0.54 mL, 6 mmol) was added and the mixture stirred for a further 16 h at r.t. The mixture was filtered and the filtrate evaporated to dryness. The residue was chromatographed on silica gel using hexanes/EtOAc (1:1) as eluent to give **11n** (0.59 g, 81%) as a white solid (mp 77.8–78 °C).

¹H NMR (CDCl₃, 300 MHz): δ = 7.27 (d, *J* = 8.5 Hz, 2 H), 6.88 (d, *J* = 8.5 Hz, 2 H), 4.60 (d, *J* = 4.4 Hz, 2 H), 4.11 (t, *J* = 5.8 Hz, 2 H), 3.85 (br q, *J* = 5.5 Hz, 2 H), 2.04 (q, *J* = 5.8 Hz, 2 H), 1.90 (br t, *J* = 5.1 Hz, 1 H), 1.77 (br t, *J* = 5.5 Hz, 1 H).

¹³C NMR (CDCl₃, 62 MHz): δ = 133.3, 128.6, 114.5, 65.7, 64.9, 60.3, 31.9.

MS (EI): *m/z* (%) = 182 (83, M⁺).

Anal. calcd. for C₁₀H₁₄O₃: C, 65.92; H, 7.74. Found: C, 66.30; H, 8.09.

IR (KBr): ν = 3248, 2922, 2868, 1612, 1514, 1468, 1248, 1020 cm⁻¹.

Acknowledgement

We thank the Dirección General de Enseñanza Superior (DGES, project PM97-0074) and the University of Alcalá (019/96; E032/97) for financial support.

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Article Identifier:

1437-210X,E;2001,0,03,0382,0388,ftx,en;P038000SS.pdf