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Aerobic oxidation of substituted phenols catalysed by metal acetylacetonates in the presence of 3-methylbutanal

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Abstract

The aerobic oxidation of substituted phenols with the catalytic system $M(acac)_n$/3-methylbutanal/O_2 has been investigated. $Co(acac)_2$ and $Mn(acac)_3$ promoted the transformation of 2,6-dimethylphenol and 2,6-di-*t*-butylphenol into their corresponding diphenoquinones and benzoquinones. In the oxidation of 2,3,6-trimethylphenol, the same catalysts yielded 32–34% of the relevant biphenol. $Cu(acac)_2$ converted 2-naphthol into 1,1′-bi-2-naphthol with 84% yield. Supported Co(II) and Cu(II) complexes have also been used as heterogeneous catalysts for the oxidation of 2,6-di-*t*-butylphenol and 2-naphthol, respectively.

Keywords: Catalytic oxidation; Substituted phenols; Benzoquinones; Diphenoquinones; Biphenols

1. Introduction

The aerobic oxidation of substituted phenols constitutes a broad and interesting research topic both from the biological [1,2] and synthetic point of view [3].

Among recent examples of oxidation of substituted phenols are the synthesis of *p*-benzoquinones and diphenoquinones catalysed by supported Mo(VI) or V(V)/*t*-butylhydroperoxide systems [4].

Porphyrin complexes of Mn(III)/H_2O_2 [5], Co(babp)/O_2 (bapb = deprotonated benzoylaminopyridine) [6], $RuCl_3$/H_2O_2 [7], or heteropolyacids/H_2O_2 [8] systems have been used for the selective synthesis of *p*-benzoquinones. Moreover, supported transition metal diketonates have been tested in the aerobic oxidation of phenols to *o*-benzoquinones [9], and alumina-supported $CuSO_4$ was found to catalyse the selective synthesis of diphenoquinones [10]. More recent studies report on the use of P-Mo-V heteropolyacids in the two-phase oxidation of 2,6-dialkylphenols [11], on the use of Cu(II) supported on a NaZSM-5 zeolite for the aerobic oxidation of 2,6-di-*t*-butylphenol [12] and on the use of $MeReO_3$/H_2O_2 as oxidising system for phenols [13].

As part of our continuing interest in the potentialities of the catalytic system metal dioxygenate/aldehyde/O_2 in the homogeneous or heterogeneous oxidation of organic substrates [14–18], we deemed it worthwhile to study the oxidation of substituted phenols, with an eye both to the selectivity of the process, and to the possibility of heterogenisation of the system using suitable hybrid catalysts.

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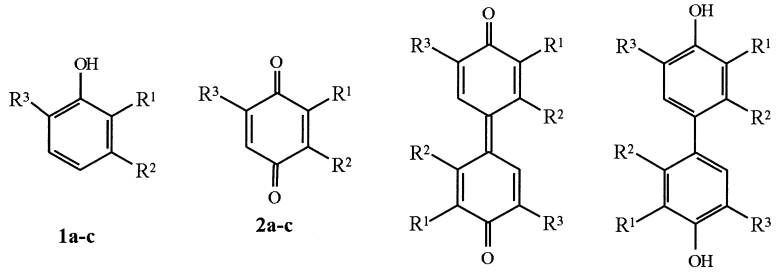


Scheme 1.

2. Results and discussion

In Scheme 1 are depicted some of the possible oxidation products derived from oxidation phenols of unsubstituted in the *para* position. 2,6-Dimethylphenol **1a** was exposed to 1 atm dioxygen at room temperature or 40°C, in the presence of 3-methylbutanal and catalytic amount of a metal acetylacetonate, the typical ratio $M(acac)_n$/substrate being 1/30. Blank tests carried out in the absence of any metal complex resulted in no reaction.

$Cu(acac)_2$, $Ni(acac)_2$, $Fe(acac)_3$ and $Pd(acac)_2$ exhibited no catalytic activity towards the oxidation of **1a**, whereas $Co(acac)_2$ and $Mn(acac)_3$ facilitated the oxidation of the substrate to the corresponding diphenoquinone **3a** derived from oxidative coupling. The conversion was in both cases almost quantitative (98% after 72 h in the case of $Co(acac)_2$ and 95% after 48 h in the case of $Mn(acac)_3$), and the isolated yields in **3a** were 70% in the case of $Co(acac)_2$ and 65% in the case of $Mn(acac)_3$ (entries 1 and 2 of Table 1). Carrying out the reactions at 40°C resulted in higher activity but lowered selectivity in both cases. After 24 h reaction, $Co(acac)_2$ gave 50% yield at 92% conversion (entry 3), whereas $Mn(acac)_3$ gave 53% yield at 97% conversion (entry 4).

When 2,6-di-*t*-butylphenol **1b** was submitted to oxidative conditions at room temperature in the absence of any metal catalyst, a very slow reaction occurred that reached only 9% conversion after 48 h (yields: **2b** = 1%, **3b** = 7%, **4b** = 1%).

Figs. 1 and 2 report the time course of $Co(acac)_2$- and $Mn(acac)_3$-catalysed oxidations of **1b** carried out at 40°C. In the case of $Co(acac)_2$, the reaction reached quantitative conversion after 48 h. Within the first 24 h, the reaction proceeds with the contemporaneous formation of comparable amounts of benzoquinone **2b** and diphenoquinone **3b**. After 48 h, the mixture contained 61% of **2b** and 2% of **3b**, along with little amount (<5%) of diphenol **4b** and other unidentified products. This behaviour can be rationalised invoking an oxidative cleavage of the initially formed **3b** into **2b**.

The $Mn(acac)_3$-catalysed oxidation gave lower conversion of the substrate (80% after 48 h) and afforded **3b** as the major product (47%, entry 4 of Table 2).

Table 1
Oxidation of 2,6-dimethyphenol **1a**[a]

Entry	Catalyst	3-Methylbutanal (mmol)	Temperature (°C)	Time (h)	Conversion (%)	Yield in **3a** (%)
1	$Co(acac)_2$	1.8 × 4	21	72	98	70
2	$Mn(acac)_3$	1.8 × 3	21	48	95	65
3	$Co(acac)_2$	1.8 × 2	40	24	92	50
4	$Mn(acac)_3$	1.8 × 2	40	24	97	53

[a] Oxidation of 2,6-dimethyphenol **1a** (1.21 mmol) in the presence of 3.3% catalyst (4.0×10^{-2} mmol), 3-methylbutanal (added in successive 1.8 mmol portions at 0, 9, 25, 49 h), O_2 (1 atm); solvent: 1,2-dichloroethane (4.5 ml).

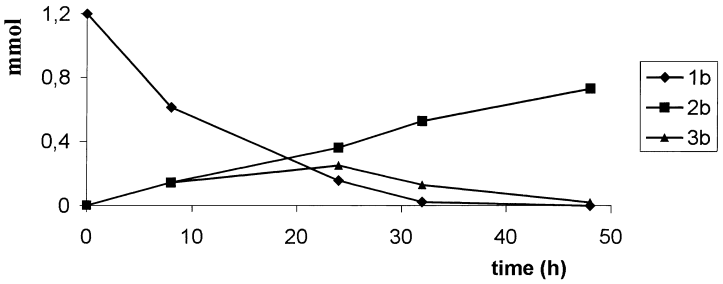


Fig. 1. Time course of the $Co(acac)_2$-catalysed oxidation of **1b** in the presence of 3-methylbutanal added in 3×1.8 mmol portions at 0, 8 and 24 h at $p_{O_2} = 1$ atm and $T = 40^\circ$C.

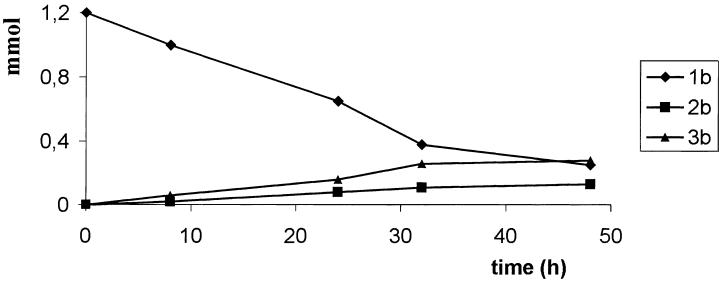


Fig. 2. Time course of the $Mn(acac)_3$-catalysed oxidation of **1b** in the presence of 3-methylbutanal added in 3×1.8 mmol portions at 0, 8 and 24 h at $p_{O_2} = 1$ atm and $T = 40^\circ$C.

It is apparent from the time course depicted in Fig. 2 that oxidative cleavage of **3b** plays only a minor role in this case.

The reaction carried out in the presence of $Fe(acac)_3$ completely converted the starting phenol in 48 h, affording 40% yield of diphenoquinone **3b** and 25% of quinone **2b** (entry 5 of Table 2).

Table 2
Oxidation of 2,6-di-*t*-butylphenol **1b**[a]

Entry	Catalyst	Conversion (%)	Yield in **3b** (%)	Yield in **2b** (%)
1	None	9	7	1
2	$Co(acac)_2$	100	2	61
3	Co-polymer	96	37	31
4	$Mn(acac)_3$	80	47	10
5	$Fe(acac)_3$	99	40	25
6	$Cu(acac)_2$	74	35	12
7	$Pd(acac)_2$	43	15	6

[a] Oxidation of 2,6-di-*t*-butylphenol **1b** (1.21 mmol) at 40°C in the presence of 3.3% catalyst (4.0×10^{-2} mmol of metal), 3-methylbutanal (1.8×3 mmol added at 0, 9, 24 h), O_2 (1 atm); solvent: 1,2-dichloroethane (4.5 ml); reaction time = 48 h.

Conversions as high as 74 and 43%, respectively, were observed in $Cu(acac)_2$- and $Pd(acac)_2$-catalysed oxidations (entries 6 and 7 of Table 2), however, these reactions were also rather unselective.

In order to transfer to heterogeneous scale the best results obtained in the oxidation of di-*t*-butylphenol we have carried out a reaction using a heterogeneous analogue of $Co(acac)_2$ the copolymer of $Co(AAEMA)_2$ ($AAEMA^-$ = deprotonated form of 2-(acetoacetoxy)ethyl methacrylate) with suitable acrylamides as supported catalyst [19]. This reaction was slightly slower with respect to that performed with $Co(acac)_2$. Indeed the conversion of **1b** was 96% after 48 h (Fig. 3), and the selectivity towards **2b** and **3b** were 31 and 37%, respectively, (entry 3 of Table 2).

The reaction of **1c** carried out in the absence of any metal catalyst gave 26% conversion after 48 h at 21°C (entry 1 of Table 3). However, this reaction resulted in an unselective oxidation leading to unidentified products. Only 4% of **4c** and negligible amount of **2c** was detected in the reaction mixture. Similar results were

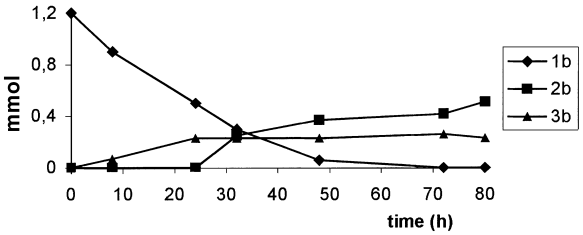


Fig. 3. Time course of the Co-polymer-catalysed oxidation of **1b** in the presence of 3-methylbutanal added in 3×1.8 mmol portions at 0, 8 and 24 h at $p_{O_2} = 1$ atm and $T = 40°C$.

Table 3
Oxidation of 2,3,6-trimethylphenol **1c**[a]

Entry	Catalyst	Conversion (%)	Yield in **4c** (%)
1	None	26	4
2	$Co(acac)_2$	66	34
3	$Mn(acac)_3$	85	32
4	$Fe(acac)_3$	100	6
5	$Cu(acac)_2$	29	8
6	$Pd(acac)_2$	32	8

[a] Oxidation of 2,3,6-trimethylphenol **1c** (1.21 mmol) at 21°C in the presence of 3.3% catalyst (4.0×10^{-2} mmol), 3-methylbutanal (1.8×3 mmol added at 0, 9, 24 h), O_2 (1 atm); solvent: 1,2-dichloroethane (4.5 ml); reaction time = 48 h.

obtained using $Cu(acac)_2$ and $Pd(acac)_2$ (entries 5 and 6 of Table 3).

$Co(acac)_2$- and $Mn(acac)_3$-catalysed reactions gave higher conversions (66 and 85%, respectively, after 48 h), and the reaction mixtures contained 34 and 32%, respectively, of the corresponding biphenol **4c** (entries 2 and 3 of Table 3). Among the by-products observed were the quinone **2c** and the coupling product of the phenoxy radical with the substrate (2,3,6-trimethyl, 4-(2,3,6-trimethyl-phenoxy)-phenol), that was identified by its MS fragmentation pattern.

When these reactions were carried out at 40°C, the increased reaction rate went along with a drop in selectivity. In the case of $Co(acac)_2$, 11% of **4c** at 71% conversion was obtained after 22 h; in the case of $Mn(acac)_3$, 15% of **4c** at 97% conversion was achieved after 22 h.

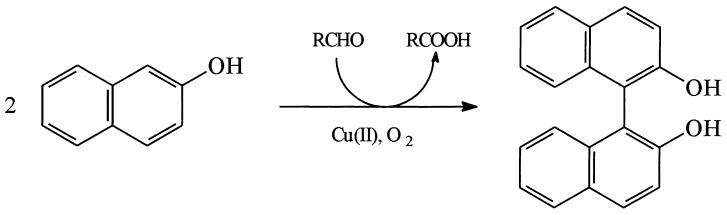
The conversion of the substrate using $Fe(acac)_3$ as the metal catalyst was quantitatively analysed after 48 h, but a complex mixture of unidentified products was obtained, among which only 6% of **4c** could be detected (entry 4 of Table 3).

2-Naphthol **1d** is a very interesting substrate to test, because of the well known application of its dimer (1,1′)binaphthalenyl-2,2′-diol **4d** and its derivatives in chirality induction [20,21]. This prompted several studies on the homogeneous and heterogeneous catalytic dimerisation of **1d** [10,22].

In this regard, we explored the behaviour of the system $M(acac)_n$/aldehyde/O_2 towards the oxidative coupling of **1d** (Scheme 2).

$Fe(acac)_3$, $Ni(acac)_2$ and $Pd(acac)_2$ gave unsatisfactory results in terms of both activity and selectivity. The maximum conversion was that obtained in the case of $Pd(acac)_2$ and was as high as 33% after 48 h. $Co(acac)_2$ and $Mn(acac)_3$ gave quantitative conversions after 29 h, but suffered from very low selectivities (<10%) towards the binaphthol **4d**. Better results were obtained using Cu(II)-based catalysts both in homogeneous and in heterogeneous phase (Table 4). The reactions carried out at 21°C with $Cu(acac)_2$ and with the copolymer (in Table 4 referred to as Cu-polymer) obtained by thermal copolymerisation of $Cu(AAEMA)_2$ with *N*,*N*-dimetylacrylamide and *N*,*N*′-methylenebisacrylamide gave 70 and 46% conversions, respectively. The relevant yields in **4d** were 56 and 33%.

Carrying out the reaction at 30°C had a beneficial effect on selectivity in the case of the $Cu(acac)_2$-



Scheme 2.

Table 4
Oxidation of **1d**[a]

Entry	Catalyst	Temperature (°C)	Conversion (%)	Yield in **4d** (%)
1	$Cu(acac)_2$	21	70	56
2	$Cu(acac)_2$	30	90	84
3	Cu-pol	21	46	33
4	Cu-pol	30	53	45

[a] Oxidation of **1d** (1.21 mmol) in the presence of 3.3% catalyst (4.0×10^{-2} mmol of $Cu(acac)_2$ or of supported copper), 3-methylbutanal (1.8×3 mmol added at 0, 9, 24 h), O_2 (1 atm); solvent: 1,2-dichloroethane (4.5 ml); reaction time = 48 h.

catalysed oxidation (entry 2). Disappointingly, only 45% yield in **4d** was obtained when Cu-polymer was used as metal catalyst.

3. Experimental

3.1. General procedures

2,6-Dimethylphenol **1a**, 2,6-di-*t*-butylphenol **1b**, 2,3,6-trimethylphenol **1c** and 2-naphthol were purchased from Fluka and used as received. 3-Methylbutanal, *n*-dodecane, $Co(acac)_2$, $Cu(acac)_2$, $Fe(acac)_3$, $Ni(acac)_2$, $Mn(acac)_2$ and $Pd(acac)_2$ were purchased from Aldrich. $Cu(AAEMA)_2$ [23] was synthesised by reacting $Cu(NO_3)_2$ and KAAEMA in 1:2.1 ratio in water following the procedure described for the synthesis of $Co(AAEMA)_2$ [24].

Chromatographic analyses were carried out on Hewlett-Packard 6890 instruments using HP-1 methyl-siloxane (60.0 m × 250 μm × 1.00 μm) or HP-5 phenyl-methyl siloxane 30.0 m × 320 μm × 0.25 μm capillary columns (injector temperature 280°C, FID temperature 280°C, carrier: nitrogen or helium). GCMS data (EI, 70 eV) were acquired on the HP 6890 instrument using HP-5MS 5% phenyl methyl siloxane 30.0 m × 250 μm × 0.25 μm capillary column coupled with a mass spectrometer HP 5973 (injector temperature 280°C, carrier: helium). IR spectra were recorded on a Perkin-Elmer 681 instrument. NMR spectra were recorded on a Bruker AM 500 spectrometer using $CDCl_3$ or toluene-d_8 as solvent. Chemical shifts for ^1H and ^{13}C are reported relative to solvent resonance and are given in ppm.

The formation of the polyphenylether was ruled out for all reactions on the basis of the lack of precipitation after addition of a five-fold excess methanol/0.3% HCl [25]. Conversions and yields were calculated using *n*-dodecane as internal standard. The yields for **3b** were assessed by direct weighting. Conversions were calculated as moles of converted substrate per mole of starting phenol. The yields reported in Tables 1–4 represent the amount of starting phenol that is converted into the relevant product and are obtained as: moles of benzoquinone/moles of starting phenol in the case of **2a**–**c**, moles of diphenoquinone × 2/moles of starting phenol in the case of **3a**–**c**, moles of diphenol × 2/moles of starting phenol in the case of **4a**–**d**, the factor 2 taking into due account the dimerisation of the substrate. Oxidation products have been identified by comparison of their masses, IR and NMR spectra with those reported in the literature.

3.2. Oxidation of **1*a***

A 250 cm^3 round flask was charged with **1a** (147 mg, 1.21 mmol), the metal acetylacetonate (0.040 mmol), 3-methylbutanal (added as specified in Table 1) and *n*-dodecane as internal standard in 1,2-dichloroethane (4.5 ml), and continuously stirred under dioxygen ($p = 1$ atm) at the desired temperature, and monitored by GLC and GCMS. When the reaction reached completion, 3 ml CH_3OH was added and the resulting red suspension was filtered, washed with methanol and dried under vacuum affording pure **3a** as red needles.

3.3. 3,5,3′,5′-tetramethyl-bicyclohexylidene-2,5,2′,5′-tetraene-4,4′-dione **3*a***

^1H NMR ($CDCl_3$): $\delta = 2.13$ (s, 12H), 7.70 (s, 4H). ^{13}C NMR ($CDCl_3$): $\delta = 17.15$, 129.64, 135.64, 138.94, 187.32. IR (KBr, cm^{-1}): 3089 (w), 3054 (w), 3019 (w), 2943 (m), 2915 (m), 1638 (s), 1592 (vs), 1562 (vs), 1426 (m), 1381 (m), 1371 (m), 1354 (m), 1218 (s), 1048 (s), 914 (s), 829 (m), 776 (m), 475 (s).

3.4. Oxidation of **1*b***

A 250 cm^3 round flask was charged with **1b** (250 mg, 1.21 mmol), the metal catalyst (0.040 mmol of metal acetylacetonate or of supported cobalt), 3-methylbutanal (added as specified in Table 2) and *n*-dodecane as internal standard in 1,2-dichloroethane

(4.5 ml), and continuously stirred under dioxygen ($p = 1$ atm) at 40°C. The reactions were monitored by GLC and GCMS. Pure **2b** and **3b** could be isolated by silica gel chromatography of the crude after treatment with aqueous Na_2CO_3 (eluant: petroleum ether bp 40–60°C/toluene 8/2). The diphenol **4b** was identified on the basis of its MS fragmentation.

3.5. 2,6-di-t-butyl-(1,4)benzoquinone **2b**

^{1}H NMR ($CDCl_3$): $\delta = 1.24$ (s, 18H), 6.47 (s, 2H). ^{13}C NMR ($CDCl_3$): 29.36, 35.56, 130.13, 157.90, 187.79, 189.11. IR (KBr, cm^{-1}): 3007–2969 (m to s), 1655 (vs), 1598 (m), 1455 (m), 1364 (m), 1317 (m), 1243 (m), 1154 (w), 1073 (w), 1024 (w), 921 (m), 879 (m), 460 (w). MS (*m*/*z* (I_{rel}%)): 41 (26), 43 (11), 67 (21), 77 (12), 79 (10), 91 (22), 95 (13), 105 (14), 107 (13), 119 (12), 135 (36), 136 (16), 147 (13), 149 (35), 159 (10), 163 (86), 164 (16), 177 (97), 178 (13), 191 (100), 192 (22), 205 (27), 220 (64, M^+), 221 (10).

3.6. 3,5,3′,5′-tetra-t-butyl-bicyclohexylidene-2,5, 2′,5′-tetraene-4,4′-dione **3b**

^{1}H NMR ($CDCl_3$): $\delta = 1.34$ (s, 36H), 7.68 (s, 4H). ^{13}C{^{1}H} NMR ($CDCl_3$): $\delta = 29.56$, 36.00, 125.99, 136.12, 150.41, 186.45. IR (KBr, cm^{-1}): 2960 (s), 1638 (s), 1606 (vs), 1566 (s), 1481 (s), 1459 (s), 1383 (m), 1361 (vs), 1338 (m), 1291 (m), 1261 (s), 1089 (vs), 1040 (s), 898 (vs), 883 (s), 842 (s), 515 (m). MS: (*m*/*z* (I_{rel}%)): 41 (11), 57 (48), 239 (11), 253 (12), 267 (10), 281 (15), 295 (28), 296 (13), 309 (42), 310 (15), 311 (11), 323 (17), 324 (16), 337 (18), 338 (14), 351 (69), 352 (36), 353 (14), 365 (12), 366 (35), 393 (37), 394 (11), 408 (100, M^+), 409 (31).

3.7. 3,5,3′,5′-tetra-tertbutyl-biphenyl-4,4′-diol **4b**

MS (*m*/*z* (I_{rel}%)): 57 (13), 162 (11), 190 (14), 395 (18), 408 (14), 410 (M^+, 100), 411 (31).

3.8. Oxidation of **1c**

A 250 cm^3 round flask was charged with **1c** (164 mg, 1.21 mmol), the metal acetylacetonate (0.040 mmol), 3-methylbutanal (added as specified in Table 3) and *n*-dodecane as internal standard in 1,2-dichloroethane (4.5 ml), and continuously stirred under dioxygen ($p = 1$ atm) at the desired temperature. The reactions were monitored by GLC and GCMS. Pure **4c** could be isolated by silica gel chromatography of the crude after treatment with aqueous Na_2CO_3 (eluant: petroleum ether bp 40–60°C/ethyl acetate 8/2).

3.9. 2,3,5,2′,3′,5′-hexamethyl-biphenyl-4,4′-diol **4c**

^{1}H NMR ($CDCl_3$): $\delta = 2.44$ (s, 6H), 2.73 (s, 6H), 2.74 (s, 6H), 7.77 (s, 2H). ^{13}C NMR ($CDCl_3$): $\delta =$ 12.22, 15.82, 16.87, 119.39, 121.57, 133.79, 134.62, 150.70. IR (KBr, cm^{-1}): 3473 (s), 1469 (s), 1371 (m), 1216 (s), 1003 (m), 854 (m). MS (*m*/*z* (I_{rel}%)): 225 (20), 237 (8), 238 (7), 239 (14), 240 (815), 241 (22), 254 (9), 255 (54), 256 (10), 269 (13), 270 (100, M^+), 271 (30).

3.10. Oxidation of **1d**

A 250 cm^3 round flask was charged with **1d** (174 mg, 1.21 mmol), the metal catalyst (0.040 mmol of metal acetylacetonate or of supported copper), 3-methylbutanal (added as specified in Table 4) and *n*-dodecane as internal standard in 1,2-dichloroethane (4.5 ml), and continuously stirred under dioxygen ($p = 1$ atm) at the desired temperature. The reactions were monitored by GLC and GCMS.

3.11. Copolymerisation of Cu(AAEMA)$_2$

$Cu(AAEMA)_2$ (0.105 g), *N*,*N*′-methylenebisacrylamide (0.093 g) and dimethylacrylamide (2.217 ml) was dissolved in acetone/*N*,*N*-dimethylformamide (4 + 4 ml) and heated at 60°C for 5 h. The green solid which formed in the reaction vessel was filtered, washed with acetone and diethylether, and dried under vacuum.

Yield: 0.72 g of polymer. Analysis: Cu: 0.86; C: 50.94; H: 8.60; N: 11.19 %. IR (Nujol, cm^{-1}): 1720, 1638, 1501, 1263, 1146, 987.

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