Oxidation of allyl and benzyl ethers by 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ)

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Benzylic and allylic ethers are oxidized by 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) to give the corresponding carbonyl containing compounds (aldehydes and ketones) and alcohols. The reaction proceeds readily with ethers bearing activating groups. Primary ethers are further oxidized with excess DDQ giving the corresponding esters. Mechanisms for these processes are proposed, based on nuclear magnetic resonance and stoichiometric analyses.

Key words: dehydrogenation, allylic and benzylic ethers.

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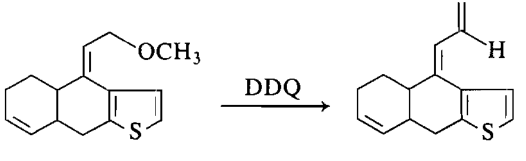
L'oxydation d'éthers benzyliques et allyliques par la dichloro-2,3 dicyano-5,6 benzoquinone (DDQ) fournit les composés carbonylés correspondants (aldéhydes et cétones) ainsi que les alcools. La réaction se produit facilement avec les éthers portant des groupements activants. Sous l'influence d'un excès de DDQ, les éthers primaires subissent une oxydation plus poussée conduisant aux esters correspondants. En se basant sur les spectres en résonance magnétique nucléaire et sur des analyses stoechiométriques, on propose des mécanismes pour ces processus.

Mots clés : déhydrogenation, éthers benzyliques et allyliques.

[Traduit par la revue]

Introduction

The use of 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) (1, 2) in the dehydrogenation of annelated cyclohexanes to give polycyclic benzenoid derivatives is the method of choice principally because of the relatively mild conditions compared to other dehydrogenation agents. The oxidation proceeds by a mechanism thought to involve initial hydride abstraction (1*b*, *c*) to generate a stabilized benzylic or allylic cation, followed by loss of a proton and concomitant formation of reduced 2,3-dichloro-5,6-dicyanohydroquinone, which usually precipitates out of solution in nonpolar solvents. Evidence for such an ionic mechanism has been obtained from trapping of the intermediate carbocations with nucleophilic solvents such as water (3) and methanol (4). In our investigations on the preparation of polynuclear aromatic hydrocarbons incorporating thiophene rings (5) we observed that, upon attempted dehydrogenation of the tetrahydro naphthothiophene **1** with DDQ, the only product formed was the unsaturated aldehyde **2**. Although oxidations of allylic alcohols with DDQ have been observed (6, 7), the conversion of **1** and **2** by DDQ represents formally a demethanation reaction that to our knowledge is unprecedented in quinone chemistry. Recently Yonemitsu and co-workers (8, 9) have shown that the 4-methoxybenzyl group can be used as a protecting group for hydroxy functions with selective deprotection taking place in the presence of DDQ and water. However, no mechanistic details were provided in these studies. To investigate the scope and limitation of this unusual oxidation we carried out a systematic study of the DDQ reaction with representative benzylic and allylic ethers.



1 **2**

Results and discussion

In addition to the allyl methyl ether **1** used in the study, the following allylic and benzylic ethers were investigated: benzyl methyl ether (**3**), benzyl ethyl ether (**4**), *trans*-cinnamyl methyl ether (**5***a*), *trans*-cinnamyl benzoate (**5***b*), *trans*-cinnamyl benzyl ether (**5***c*), *trans*-cinnamyl trityl ether (**5***d*), 1,1-phenylmethoxyethane (**6**), methyl 4-methoxy-2-butenoate (**7**), thiophenoxymethyl methyl ether (**8**), 1,2-phenylmethoxyethane (**9**), *p*-nitrobenzyl methyl ether (**10**), and 3-methoxycyclohexene (**11**). The reactions were carried out in toluene solution heated to reflux or in carbon tetrachloride at room temperature. The details of individual reactions are tabulated in Table 1. All of the products were compared with known compounds. Although these reactions formally represent oxidative dealkylations, the alkyl group is transformed to its alcohol in the products as seen from the reactions of **5***b*–*d*. Although the presence of methanol could be confirmed by nmr spectroscopy from the reactions of the methyl ethers, its isolation was difficult due to losses in the work-up procedures. It is noteworthy that the oxidations proceed with allyl and benzyl ethers that do not possess electron-withdrawing groups, as can be seen from the absence of oxidation products in the cases of **7** and **10**.

Furthermore, the inertness of **7**, **9**, and **10** to oxidation suggests that the initial hydride abstraction and formation of a carbocation is rate determining. To shed some light on the mechanism and establish the fate of the methyl group during the oxidation of the methyl allyl/benzyl ethers with DDQ we carried out a systematic nmr investigation of ether **5***a*, which was found to be most reactive in this series and gave smooth conversion at room temperature.

The ^{1}H nmr spectrum of a $CCl_4/CDCl_3$ solution containing *trans*-cinnamyl methyl ether (20 mM) and DDQ (20 mM) was observed at room temperature. The spectrum after 1 h showed a small amount of cinnamaldehyde (17%) in addition to signals corresponding to an acetal **13**. The presence of cinnamaldehyde was significantly diminished upon the addition of ground and freshly activated 4 Å molecular sieves.

The assignment of the mixed acetal was based on the splitting pattern of the acetal and vinylic protons at 5.8 ($J = 5.8$ Hz)

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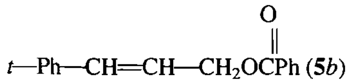
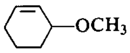
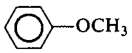
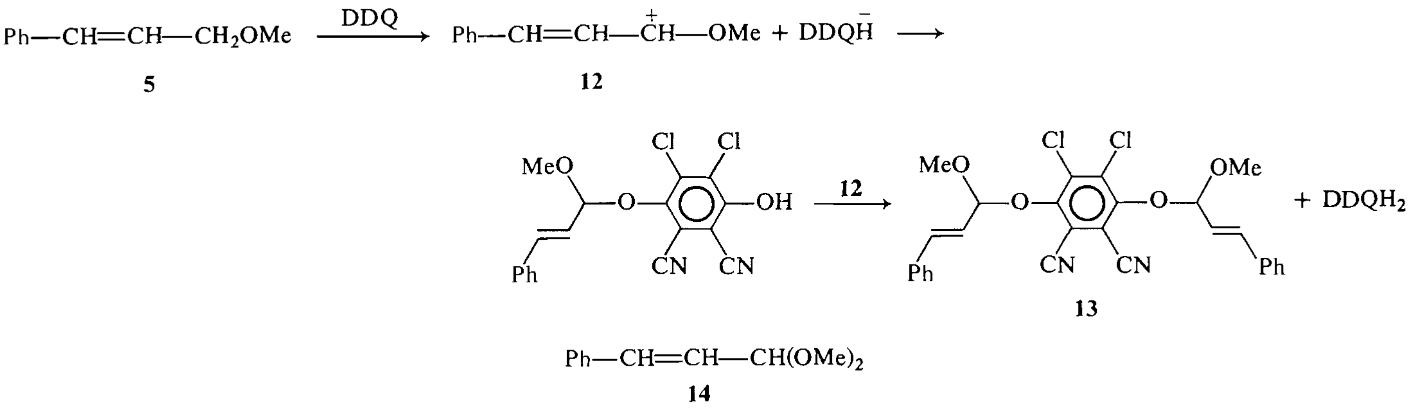
TABLE 1. Results of DDQ oxidation of allyl/benzyl methyl ethers

$$\text{Z—CH}_2\text{—OCH}_3 \xrightarrow[\text{(2) H}_2\text{O}]{\text{(1) DDQ/PhMe, }\Delta} \text{Z—CHO}$$

Z = aryl or vinyl substituent

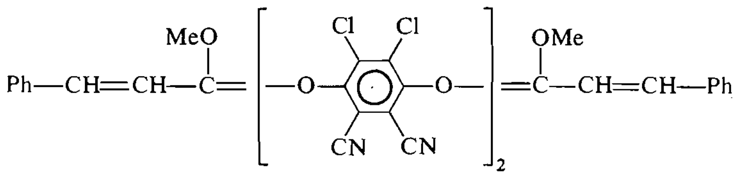
Ether	Reaction conditions	Product (yield)
1	Reflux, 2 h	**2** (52)[a]
Ph—CH_2OCH_3 (**3**)	Reflux, 5 h	Ph—CHO (75)[e,f]
Ph—CH_2—O—CH_2CH_3 (**4**)	Reflux, 5 h	Ph—CHO (37)[b]
t—Ph—CH=CH_2OCH_3 (**5*a***)	CCl_4/r.t./3 h	*t*—Ph—CH=CH—CHO (85)[c,e,f]
t—Ph—CH=CH—CH_2OCH_3 (**5*a***)	Chloranil/PhMe/reflux, 4 h	*t*—Ph—CH=CH—CHO (54)[c]
 $t\text{—Ph—CH=CH—CH}_2\text{O}\overset{\overset{\text{O}}{\|}}{\text{C}}\text{Ph}$ (**5*b***)	CCl_4/r.t./3 h	Cinnamaldehyde (61)[e,f] + $PhCO_2H$ (72)[e,f]
t—Ph—CH=CH—CH_2OCH_2Ph (**5*c***)	CCl_4/r.t./3 h	Cinnamaldehyde (68) + $PhCH_2OH$ (53)[e,f]
t—Ph—CH=CH—$CH_2OC(Ph)_3$ (**5*d***)	CCl_4/r.t./3 h	Cinnamaldehyde (69) + $(Ph)_3COH$ (87)[e,f,g]
Ph·CH(CH_3)OCH_3 (**6**)	Reflux, 22 h	Ph—CO—CH_3 (58)[d,e,f]
Ph·CH(CH_3)OCH_3 (**6**)	1,4—Dioxan/reflux, 5 h	N.R.
$MeOCH_2CH$=$CHCO_2Me$ (**7**)	Reflux, 20 h	N.R.
Ph—S—CH_2OCH_3 (**8**)	Reflux, 22 h	N.R.
Ph—CH_2CH_2—OCH_3 (**9**)	Reflux, 5 h	N.R.
p—NO_2—C_6H_4—CH_2OCH_3 (**10**)	Reflux, 3 h	N.R.
 (**11**)	Reflux, 3 h	 (50)[c]

[a] Structure tentatively assigned on the basis of ir, nmr, and mass spectral data.
[b] Product aldehyde isolated as bisulfite addition compound.
[c] Yield estimated by ^1H nmr spectroscopy after aqueous work-up.
[d] Conversion was 77% based on unreacted starting material.
[e] Product identified by comparison of ^1H nmr and ir spectra with those of an authentic sample.
[f] Product identified by tlc comparison to authentic standard.
[g] Recrystallized product had mp identical with that reported in the literature.



SCHEME 1

(H—C—O), 6.1, and 6.7 ppm respectively, and comparison of these with acetal **14** prepared independently. The methoxy protons of **13** appeared as a pair of singlets at 3.35 and 3.40 ppm. The relative integration of the methoxyl to acetal proton signals was approximately 3:1. Comparison of the spectral data of the reaction mixture with authentic **14** rules out the presence of the dimethyl acetal **14**. Furthermore, only one half of the expected reduced $DDQH_2$ precipitated from solution at this point in time, although the reduction product $DDQH_2$ is insoluble in carbon tetrachloride. This observation, along with the formation of a major product containing an acetal proton as seen by ^1H nmr, suggests that one half equivalent of DDQ is covalently bound to the oxidized substrate. Attempted isolation and purification of acetal **13** by chromatography led to its hydrolysis to *trans*-cinnamaldehyde and $DDQH_2$ (~½ equiv.). Increasing the initial concentration of DDQ to 1.8 equivalents (36 mM) resulted in the formation of methyl *trans*-cinnamate in addition to cinnamaldehyde. (This ester was also obtained in 12% yield as a by-product from the oxidation of the ether **5*a*** with 1.1 equivalents of DDQ.) Following this reaction mixture by ^1H nmr spectroscopy, we observed another transient with peaks at 3.24 ppm (OCH_3) and an AB pattern at 5.9 and 6.9 ppm. This



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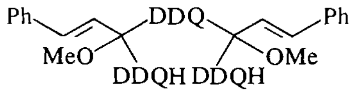

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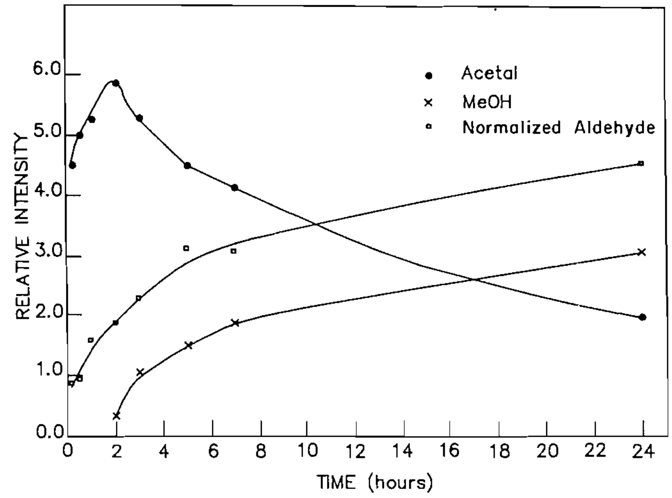


FIG. 1. Reaction profile for DDQ/ether 5*a* = 1:1 equiv.

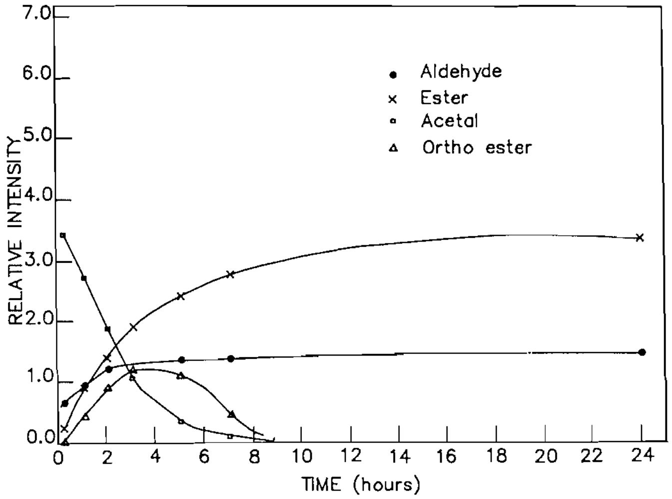


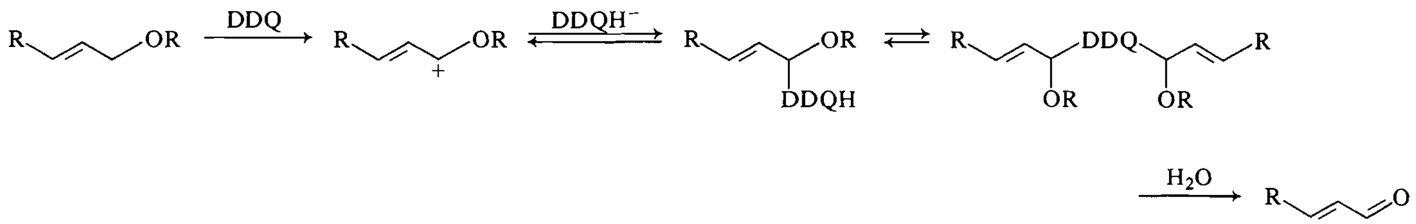
FIG. 2. Reaction profile for DDQ/ether 5*a* = 1.8:1 equiv.

transient has been tentatively assigned the structure of the ortho ester **15**. No signal associated with an acetal proton was observed in this case. The relative integration of the methoxy signal (3.43) to the vinyl signal (AB 5.9 ppm or 6.9 ppm) was approximately 3;1. These data do not preclude structures such as **16** as the transient species although the OH signals associated with **16** were not discernible in the ^{1}H nmr spectrum.

This transient upon prolonged reaction (and, ostensibly, hydrolysis) gave methyl cinnamate. Kinetic profiles based on nmr time-resolved spectra of the reaction mixtures containing 1:1 and 1.8:1 molar ratios of DDQ and ether **5** are shown in Figs. 1 and 2 respectively. It can be seen in Fig. 1 that the concentration of the acetal **13** peaks at about 2 h and then decreases with accompanying increase in formation of both methanol and cinnamaldehyde. In Fig. 2 the concentration of acetal **13** peaks earlier and decreases more rapidly than in Fig. 1. At the same time the transient associated with the ortho ester **15** gradually increases and peaks at 3.5 h before diminishing while the aldehyde and ester concentrations level off. These observations point to two intermediates from which the aldehyde and ester are formed. The nmr spectroscopic evidence suggests that the acetal **13** and the ortho esters **15** or **16** may be precursors to cinnamaldehyde and methyl cinnamate respectively. It is clear from the spectral data that cinnamaldehyde is not oxidized by DDQ to methyl cinnamate, but DDQ is oxidizing the acetal **13** to an ortho ester. The minor amount of cinnamaldehyde formed initially is attributed to the presence of adventitious water, which could be minimized by the addition of activated molecular sieves. This is demonstrated when the addition of D_2O to the reaction mixture results in hydrolysis of the acetal **13** to cinnamaldehyde in a separate nmr experiment. The data suggest that the oxidation of allylic/benzylic ethers by DDQ proceeds by initial and rate-determining hydride abstraction of the α-carbon (Scheme 2). This is seen from the relative reactivities of ethers possessing electron-donating and -withdrawing R substituents. Electron-deficient electrophilic R groups will deactivate this process as is seen in the inertness of ethers **7** and **10**, although the lack of reactivity of **8** is somewhat surprising. Given the failure of the reaction to occur in dioxan, it may be that the formation of a π-complex between DDQ and the Lewis base renders DDQ inactive as a dehydrogenating reagent. Furthermore, the stabilization of the resultant cation is crucial, as is observed in the unreactive nature of ether **9**. Thus oxidation of allylic or benzylic ethers by DDQ is possible, provided that no electrophilic substituent is prpesent in the conjugated chromophore and no available β-hydrogen is present. In the latter case dehydrogenation results as the dominant process, as is observed for ether **11**.

These results suggest that DDQ can be used as an alternative method for the deprotection of benzyl ethers under neutral conditions as was also shown by Yonemitsu (8, 9). Furthermore, the benzyl methyl ether function can be used as a "masked" aryl carboxaldehyde in which the methoxymethyl substituent would activate the aryl ring system and direct electrophilic substitution with a regiochemistry different from the formyl group.

These results also indicate that electron-rich allyl alcohols or



SCHEME 2

unsaturated aldehydes (via acetals) can be oxidized to aldehydes and esters with DDQ, without the competing degradative oxidation that is frequently a complication with the use of chromium(VI) reagents.

Experimental

The ^{1}H nmr spectra were recorded in CDCl$_3$ or CCl$_4$ solution with TMS as an internal reference on a Bruker AM-300 nmr spectrometer. Infrared spectra were recorded neat on NaCl discs on a Unicam SP1000 instrument, low resolution mass spectra were obtained on a VG Micromass 16F instrument, and high resolution mass spectra were obtained on a VG ZAB-E instrument.

Anhydrous CDCl$_3$ and CCl$_4$ were obtained by storing these over molecular sieves for 48 h. Toluene was dried over sodium metal. Cinnamyl methyl ether (**5**) was prepared from cinnamyl alcohol (10).

General procedure for DDQ oxidations

In a typical run cinnamyl methyl ether **5***a* (157.4 mg; 1.06 mmol) and DDQ (244.5 mg, 1.08 equiv.) were dissolved in dry toluene (20 mL) and refluxed for 1.5 h. The resulting slurry was cooled, DDQH$_2$ filtered out, and the solvent removed to afford an oil, which was flash chromatographed (silica gel, dichloromethane) to afford methyl cinnamate (20.0 mg, 12%) followed by cinnamaldehyde (112.0 mg, 80%).

(8a-H-4,4a,7,8-Tetrahydronaphtho[2,3-b]thien-9-ylidene)-acetaldehyde (2)

The methyl ether **1** (62.7 mg; 0.255 mmol) was dissolved in dry toluene (50 mL) with DDQ (127.8 mg, 2.2 equiv.) and the solution refluxed 2 h. The solution was cooled, concentrated, and flash chromatographed (silica gel, dichloromethane) to afford the aldehyde as the only product, **2** (30.6 mg, 52%); ir ν_{NaCl}:1680 (C═O) cm^{-1}; ^{1}H nmr δ:10.20, d(J = 6.0 Hz), CHO; 7.24, d(J = 5.2 Hz), H2; 7.13, d(J = 5.2 Hz), H1; 6.32, d(J = 6.0 Hz), Hα; 5.75–5.95, m, H5,6; 3.65, d(J = 7.5 Hz), 1H; 3.13, dd(J = 7.5,1.0 Hz), 1H; 2.60–2.80, m, 2H; 2.15–2.80, m, 2H; 1.65–2.00, m, 2H; ppm; ms*m/z* : 230(100), 201(84).

Nuclear magnetic resonance analysis of DDQ: ether 5a *mixtures*

The ether **5***a* and DDQ were mixed together in dry carbon tetrachloride to afford a 40 mM solution, which was diluted to 20 mM with deuteriochloroform. The ^{1}H nmr spectra were recorded at 23°C. The relative concentrations of the aldehyde, ester, methanol, acetal **13**, and ortho ester **15** were based on the height of the peaks (relative to CHCl$_3$) at δ 9.72, 3.81, 3.48, 3.36, and 3.24 ppm, respectively.

Cinnamaldehyde dimethyl acetal

This compound was prepared by the general procedure involving 2,2-dimethoxypropane (11) and had a bp identical to the known dimethyl acetal (12); ^{1}H nmr δ: 7.26–7.43, m, 5H; 6.73, d(J = 16.1 Hz), Hα; 6.15, dd(J = 16.1, 4.9 Hz), Hβ; 4.96, d(J = 4.9 Hz), Hγ; 3.38, s, OCH$_3$.

Preparation of cinnamyl trityl ether 5d *(Z-3-phenyl-1-triphenylmethoxy-2-propene)*

Cinnamyl alcohol (0.69 mL, 5.36 mmol) was dissolved in dry dichloromethane (10 mL) with trityl chloride (1.50 g, 1.0 equiv.) and lutidine (0.62 mL, 1.0 equiv.). The solution was stirred for 4 days, the solvent removed, and the residue recrystallized from petroleum ether:toluene (5:1, 10 mL) to afford the trityl ether (1.04 g, 52%); mp (pet. ether) 126.5–127°C; ir ν_{CHCl_3}: 2985, 1587, 1480, 1439 cm^{-1}; ^{1}H nmr δ: 7.2–7.6 m, 20H; 6.70, d(J = 15.9 Hz), Hα; 6.20, dt (J = 15.9, 5.0 Hz), Hβ; 3.79, d(J = 5.0 Hz), CH$_2$; ppm; ms *m/z*: 244(35), 243(100, Ph$_3$C$^+$), 183(17), 149(18). *Anal.* calcd. for C$_{28}$H$_{24}$O: C 89.31, H 6.44; found: C 89.49, H 6.78.

Acknowledgements

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