

Decomposition of vinyl ethers by alkalide K^- , $K^+(15\text{-crown-5})_2$ via organopotassium intermediates

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Abstract

The structure of vinyl ethers determines the direction of the C–O bond cleavage by alkalide K^- , $K^+(15\text{-crown-5})_2$ **1**. Highly reactive organopotassium compounds are intermediate products formed in the system containing phenyl vinyl ether, butyl vinyl ether, ethylene glycol butyl vinyl ether or triethylene glycol methyl vinyl ether. Vinylpotassium and butylpotassium react with 15-crown-5. The oxacyclic ring of the latter is opened in this case. Organopotassium ethers possessing $\text{CH}_2\text{CH}_2\text{O}$ units eliminate ethylene. It results in various potassium alkoxides. The reaction of **1** with butyl vinyl ether occurs very slow as compared to other vinyl ethers and most of other reagents used till now.

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Keywords: Vinyl ethers; Alkalide; Potassium anions; Organopotassium intermediates

1. Introduction

Alkalides, i.e. salts containing alkali metal anions and complexed metal cations are excellent electron-transfer reagents for the ether cleavage. Fish et al. [1] observed the regioselectivity of the ether bond scission in aromatic ethers using K^- , $K^+(18\text{-crown-6})$. Then, it was found that the regioselective cleavage of the alkyl-oxygen bond occurred also in methyl phenyl ether, ethyl phenyl ether [2], and allyl phenyl ether [3] in their reactions with K^- , $K^+(15\text{-crown-5})_2$ **1**. It was shown in further works that various cyclic ethers as monosubstituted oxiranes [3–9], oxetane [10–12] and tetrahydrofuran [13,14] can also be opened by alkalides. The most interesting seemed to be the reaction involving oxiranes. The oxacyclic ring was opened in the α - [8,9] or in the β -position [3–6,9], i.e.

between the substituted carbon atom and oxygen or between the CH_2 group and oxygen, respectively. The direction of the ether bond cleavage depended on the kind of substituent. Unexpectedly, the scission of one of the linear ether bonds was found in some oxiranes possessing an oxygen atom in the substituent [7]. The highly strained oxirane ring remained undisturbed. The decomposition of cyclic ethers in a larger ring, e.g., 18-crown-6 [13], 15-crown-5 [14], and their monocyclohexano- and dicyclohexano-derivatives [15] was observed under the influence of potassium anions in tetrahydrofuran solution.

The use of alkalides for the ether bond cleavage seemed to be an important area for organometallic chemists. That led to the generation of new organopotassium compounds being intermediate products in those systems. They were generally unstable and underwent various fast reactions, e.g., with crown ether or with the substrate [16]. Some of them eliminate potassium phenoxide [5] or unsaturated hydrocarbons, e.g.,

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ethylene [12,14] or cyclohexene [15]. Most of the reactions mentioned above had been previously not described in the literature.

However, there was a lack of information on the behaviour of vinyl ethers under the influence of alkalide. In this work, we present for the first time the results concerning this problem. Phenyl vinyl ether, butyl vinyl ether, ethylene glycol butyl vinyl ether, and triethylene glycol methyl vinyl ether as well as **1** in tetrahydrofuran solution were selected for the study.

2. Results and discussion

The reaction of **1** with phenyl vinyl ether was conducted in the first series of experiments. Two liquid products, i.e. benzyl phenyl ether **6** (in 5% yield) and tetraethylene glycol benzyl vinyl ether **7** (9%) were found in the reaction mixture after treatment with benzyl bromide as the quenching agent. Ethylene was evolved during the reaction.

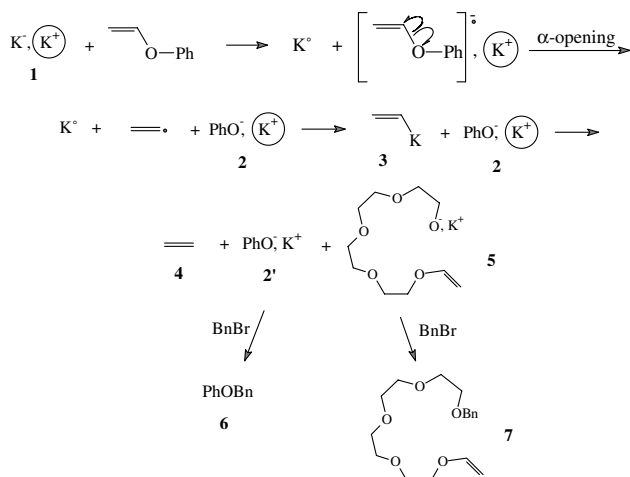
It was taken into account that the C–O bond in vinyl ethers can be opened in two ways, called in this paper as the α - or the β -opening:



The results showed that the ether bond in phenyl vinyl ether (R: Ph) was cleaved exclusively in the α -position, i.e. between the vinyl group and oxygen (Scheme 1). It was assumed that in the first step of the process K^- of **1** transfers one electron to the phenyl group giving K° and a radical anion. The latter is unstable and decomposes by the homolytic cleavage to potassium phenoxide **2** and vinyl radical. Then, K° transfers the second electron to the radical, which results in the formation of

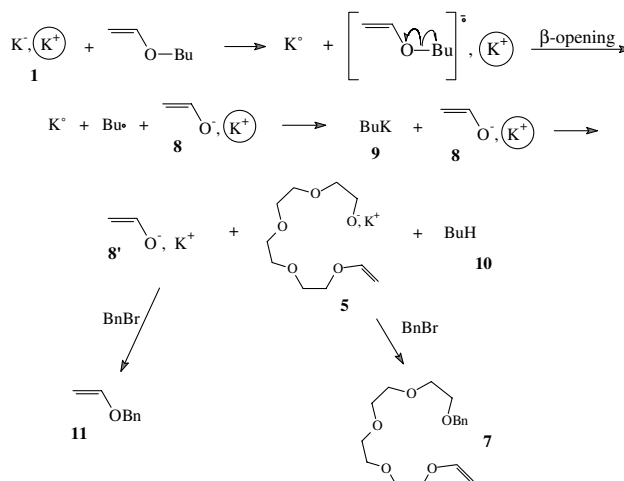
vinylpotassium **3**. It reacts with 15-crown-5 being in the complex with potassium cation of **2**. Potassium tetraethylene glycol vinyl ether **5** is the product of the reaction. A likely mechanism is that the vinylpotassium species is behaving as a base and effecting an E_2 -like elimination on the crown compound to provide the alkoxide precursor **5** to benzyl ether **7**. The cleavage of the ether bond of phenyl vinyl ether in the α -position was preferred probably due to the high resonance stability of the phenoxide anion. The same direction of the C–O bond scission was described for that ether in the presence of an excess of lithium powder and a catalytic amount of 4,4'-di-tert-butylbiphenylide (DTBB) in tetrahydrofuran at room temperature [17]. The reaction led to the vinyl lithium intermediate.

If the aromatic substituent in the vinyl ether molecule has been replaced by an aliphatic one then K^- transfers one electron to the vinyl group giving K° and the corresponding radical anion. In this case, the ether bond cleavage occurred in the β -position, i.e. between the substituent and oxygen. Such a scission was observed in butyl vinyl ether (R: Bu). Tetraethylene glycol benzyl vinyl ether **7** (8%) and benzyl vinyl ether **11** (5%) were found in the reaction mixture after treatment with benzyl bromide. Butane was evolved during the reaction. The proposed course of the process is presented in Scheme 2. The β -scission in butyl vinyl ether is probably due to the resonance stability of enolate anion of **8**. It is worth noting that potassium enolate of acetaldehyde **8'** underwent exclusively *O*-benzylation giving benzyl vinyl ether **11**. The product of *C*-benzylation, i.e. 3-phenylpropionaldehyde was not detected. The formation of *O*-alkylated product is not surprising when one considers the hard nature of this metal enolate. Sodium enolates are commonly employed to effect *O*-alkylation of enolates and presumably potassium enolates will be equally



where K° denotes $\text{K}^+(15\text{-crown-5})$; the second crown ether molecule was omitted.

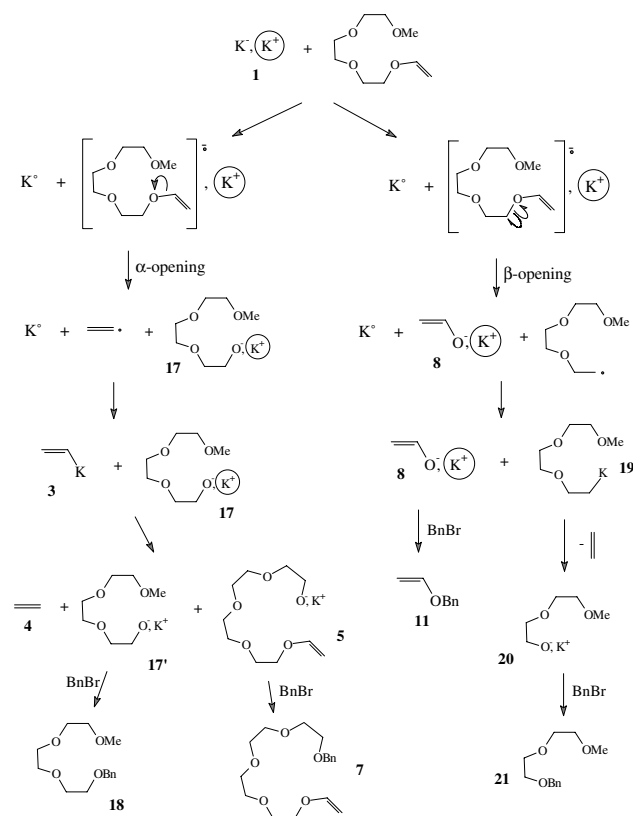
Scheme 1.



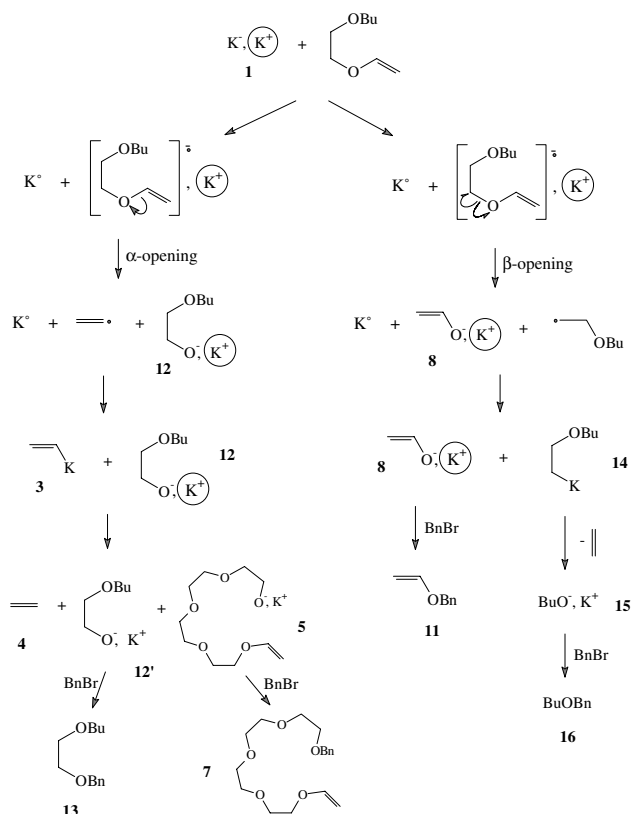
Scheme 2.

good, if not better, at achieving this regioselectivity. Unexpectedly, the reaction of **1** with butyl vinyl ether studied in the present work was rather slow. It occurred in about two hours whereas that with phenyl vinyl ether was found to be instantaneously. The end of the reaction was correlated with the discoloration of the reaction mixture indicating the absence of K^- .

Introduction of the CH_2CH_2O group or groups into the vinyl ether molecule makes the process more complex. Tetraethylene glycol benzyl vinyl ether **7** (6%), benzyl vinyl ether **11** (2%), ethylene glycol benzyl butyl ether **13** (4%), and benzyl butyl ether **16** (3%) are identified in the mixture benzylated after the reaction of **1** with ethylene glycol butyl vinyl ether (R: CH_2CH_2O Bu). On the other hand, **7** (5%), **11** (2%), triethylene glycol benzyl methyl ether **18** (5%), and diethylene glycol benzyl methyl ether **21** (4%) are found when triethylene glycol methyl vinyl ether is used (R: $(CH_2CH_2O)_2-CH_2CH_2OMe$). Ethylene is evolved in both the cases. These results suggest that the cleavage of the ether bond occurs under the influence of metal anions in the β -position and, rather unexpectedly, also in the α -position in both the ethers (Schemes 3 and 4). The β -cleavage can be connected with the resonance stability of the enolate anion **8** as well as with the stabilisation of alkylpotassium intermediates **14** and **19** by the oxygen atom or



Scheme 4.



Scheme 3.

atoms. The reason why the α -cleavage also takes place is not clear. One possible explanation bases on the difference in the number of oxygen atoms in vinyl ether molecules with and without CH_2CH_2O units. Some interaction between two or more oxygen atoms and the potassium cation, similar to those observed in glymes [18], might be responsible for such phenomenon. For example the presence of an additional oxygen atom in the vicinity of the alkoxide group changes the activity of lithium, sodium and potassium alkoxides in the polymerization of methyl methacrylate [19]. A similar effect was found in the polymerization of styrene by $H(OCH_2CH_2)_nO^-$, K^+ , where $n = 1-5$ [20]. The reaction rate, and the molecular weight of polymers were controlled by the number of oxygen atoms in the initiator molecule.

It is worth noting that in the case of ethylene glycol butyl vinyl ether and triethylene glycol methyl vinyl ether only the ether bonds neighbouring with the vinyl group are opened by potassium anions. The remaining ether bonds, i.e. CH_2-O-CH_2 and CH_2-O-Bu or CH_2-O-Me are stable.

The mechanistic study indicated that in radical anions of vinyl ethers containing the CH_2CH_2O unit or units the cleavage of the ether bond occurring between the vinyl group and oxygen is heterolytic and not homolytic

whereas in phenyl vinyl ether the homolytic α -cleavage is observed.

The organopotassium compounds formed as intermediates, i.e. **3**, **9**, **14**, and **19** underwent further fast reactions. None of their benzylated derivatives were detected in any of the systems studied. The compounds **3** and **9** reacted exclusively with the crown ether whereas **14** and **19**, i.e. containing $\text{CH}_2\text{CH}_2\text{O}$ sequences, decomposed to appropriate potassium alkoxides with the liberation of ethylene.

The yields of reaction products were low, below 10%, because the process was conducted using an excess of vinyl ethers. That allowed us to avoid side reactions between alkali and primary reaction products. At the excess of **1** further reactions take place with the participation of potassium anions. It was reported [7] that in such case some initial products were destroyed and various new compounds were observed in the reaction mixture. Therefore, that method of substrate delivery might lead to erroneous conclusions concerning the reaction mechanism.

In all the processes studied till now, the discoloration of the blue alkali solution was instantaneous after mixing with the excess of the reagent. The reaction of potassium anions was very fast at ambient temperature. The exception to this rule was observed only by the self-decomposition of potassium solutions. In that case a slow reaction of potassium anions occurred with a ligand, e.g. 18-crown-6 [13], 15-crown-5 [14], or their cyclohexano derivatives [15], and with tetrahydrofuran used as the solvent [13,14]. The next exception was found in the present work for the reaction of K^- , $\text{K}^+(15\text{-crown-5})_2$ with butyl vinyl ether. The question why that reaction occurred during about 2 h whereas the others were usually instantaneous remains unanswered.

3. Conclusions

The structure of vinyl ethers determines the direction of the ether bond cleavage by alkali K^- , $\text{K}^+(15\text{-crown-5})_2$. Vinylpotassium and butylpotassium as intermediate products open the crown ether ring being in the complex with the potassium cation. Organopotassium compounds containing the $\text{CH}_2\text{CH}_2\text{O}$ groups eliminate ethylene forming potassium alkoxides. Potassium enolate of acetaldehyde is one of the final products in most of the studied systems. It undergoes exclusively *O*-benzylation after treating with benzyl bromide, i.e. it behaves rather as an alkoxide than as an organopotassium compound. Surprisingly, the reaction of alkali with butyl vinyl ether is rather slow in comparison to most reactions of potassium anions known until now.

4. Experimental

Gaseous products were searched for by gas-chromatography (GC) with a 2.4 m long stainless steel column packed with Al_2O_3 , 0.2–0.3 mm, deactivated with 5% K_2CO_3 , in an INCO 505 gas chromatograph equipped with a flame ionization detector.

GC–MS analysis of liquids was performed on a 30-m MDN 5S capillary column in a GC Trace 2000 gas chromatograph equipped with a Finnigan Trace MS detector. Diethylene glycol was used as an internal standard for the yield measurements. The gaseous compounds were determined with a Chromatron GCHF chromatograph at ambient temperature on a 2-m metal column filled with modified alumina. NMR spectra were recorded in CDCl_3 with a Varian Unity Inova spectrometer (300 MHz for ^1H and 75 MHz for ^{13}C) at 20 °C. Chemical shifts are expressed in ppm downfield from TMS. 2D-NMR spectroscopy heteronuclear chemical shift correlation (HETCOR) and absolute value COSY were used in correlation of the lines in NMR spectra. HRMS were recorded on a Finnigan MAT 95 mass spectrometer (Finnigan MAT GmbH, Bremen, Germany).

Butyl vinyl ether, ethylene glycol butyl vinyl ether and triethylene glycol methyl vinyl ether (all Aldrich) were distilled over CaH_2 . The fractions boiling at 94, 70 °C/20 mm Hg, and 112 °C/10 mm Hg were collected, respectively. A 0.1 M K^- , $\text{K}^+(15\text{-crown-5})_2$ tetrahydrofuran solution was prepared as described in [21].

The reaction was carried out in an apparatus described in [4] under dry argon atmosphere at 20 °C. 0.1 M K^- , $\text{K}^+(15\text{-crown-5})_2$ dark blue tetrahydrofuran solution (10 mL) was introduced into 0.5 M solution of vinyl ether (10 mL) in tetrahydrofuran while mixing. After discoloration the reaction mixture was treated with benzyl bromide to transform non-volatile reaction products into the volatile compounds. The latter were then identified matching their retention times, mass spectra, and NMR spectra with those of authentic compounds.

4.1. Phenyl vinyl ether

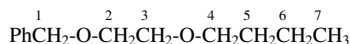
A mixture of 0.01 mol 2-bromoethyl phenyl ether, 0.01 mol 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) and tetrahydrofuran (10 mL) was vigorously stirred at 60 °C for 6 h. After cooling pentane (10 mL) was added and the mixture was left overnight at 0 °C. The salt precipitate was filtered off and washed with 10 mL of tetrahydrofuran and pentane mixture (1:1). Volatile fractions were removed by distillation and phenyl vinyl ether was obtained in practically quantitative yield (purity > 99%; GC). B.p. = 48–49 °C/12 mm Hg. Its MS data and ^1H and ^{13}C NMR spectra (CDCl_3) are presented in [22,23], respectively.

4.2. Benzyl vinyl ether (**11**)

A mixture of 0.01 mol 2-bromoethyl benzyl ether, 0.01 mol 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) and tetrahydrofuran (10 mL) was vigorously stirred at 60 °C for 8 h. After cooling pentane (10 mL) was added and the mixture was left overnight at 0 °C. The salt precipitate was filtered off and washed with 10 mL of tetrahydrofuran and pentane mixture (1:1). Volatile fractions were removed by distillation and benzyl vinyl ether was obtained in practically quantitative yield (purity > 99%; GC). B.p. = 50–51 °C/16 mm Hg. MS; *m/z* (%): 134 (M^+ , 4), 116 (7), 105 (11), 91 (100), 77 (7), 65 (30), 51 (9), 39 (11). MS data were also presented in [24], and ^1H and ^{13}C NMR (CDCl_3) spectra in [25].

4.3. Ethylene glycol benzyl butyl ether (**13**)

A mixture of 0.1 mol ethylene glycol monobutyl ether, 0.11 mol benzyl chloride, 50% NaOH (60 mL), 0.005 mol tetrabutylammonium hydrogensulfate, and benzene (50 mL) was vigorously stirred in 70–80 °C on a water bath for 3 h. After cooling, water (100 mL) and hexane (100 mL) were added. The layers were separated, the organic layer has been washed with water (3×50 mL) and dried with anhydrous MgSO_4 . Having removed hexane and benzene on a rotatory evaporator the residue has been distilled under reduced pressure. Ethylene glycol benzyl butyl ether was finally obtained in 88% yield as a novel compound (purity > 97%; GC). B.p. = 161–162 °C/2 mm Hg.

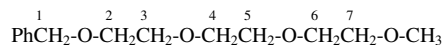


^1H NMR (CDCl_3): 7.40–7.20 (m, 5H, Ph), 4.57 (s, 2H, CH_2 -1), 3.60–3.62 (m, 4H, CH_2 -2,3), 3.47 (t, $J = 6.7$ Hz, 2H, CH_2 -4), 1.62–1.52 (m, 2H, CH_2 -5), 1.43–1.30 (m, 2H, CH_2 -6), 0.92 (t, $J = 7.2$ Hz, 3H, CH_3). ^{13}C NMR: 138.26 (C-1, Ph), 128.25 (C_{meta} , Ph), 127.64 (C_{ortho} , Ph), 127.46 (C_{para} , Ph), 73.14 (CH_2 -1), 71.14 (CH_2 -4), 70.07 (CH_2 -3), 69.35 (CH_2 -2), 31.66 (CH_2 -5), 19.21 (CH_2 -6), 13.86 (CH_3). MS, *m/z* (%): 208 (M^+ , 25), 151 (3), 134 (4), 117 (6), 107 (19), 91 (100), 73 (17), 55 (14), 57 (43), 41 (15). HRMS (EI, 70 eV) calcd. for $\text{C}_{13}\text{H}_{20}\text{O}_2$ [M^+] 208, 1463. Found 208, 1464.

4.4. Triethylene glycol benzyl methyl ether (**18**)

A mixture of 0.1 mol ethylene glycol monobutyl ether, 0.11 mol benzyl chloride, 50% NaOH (60 mL), 0.006 mol tetrabutylammonium hydrogensulfate, and benzene (50 mL) was vigorously stirred at 70–80 °C on a water bath for 3 h. After cooling, water (100 mL) and hexane (100 mL) were added. The layers were separated, the organic layer was washed with water (3×50 mL) and dried with anhydrous MgSO_4 . Having removed

hexane and benzene on a rotatory evaporator the residue was distilled under reduced pressure. Triethylene glycol benzyl methyl ether has been finally obtained in 70% yield as a novel compound (purity > 97%; GC). B.p. = 191 °C/2 mm Hg.



^1H NMR (CDCl_3): 7.40–7.20 (m, 5H, Ph), 4.56 (s, 2H, CH_2 -1), 3.70–3.60 (m, 10H, CH_2 -2,3,4,5,6), 3.48–3.56 (m, 2H, CH_2 -7), 3.36 (s, 3H, CH_3). ^{13}C NMR: 138.14 (C-1, Ph), 128.25 (C_{meta} , Ph), 127.58 (C_{ortho} , Ph), 127.47 (C_{para} , Ph), 78.06 (CH_2 -1), 71.79 (CH_2 -7), 70.49 (CH_2 -2,3,4), 70.39 (CH_2 -5), 69.27 (CH_2 -6), 58.88 (CH_3). MS, *m/z* (%): 254 (M^+ , 2), 195 (2), 148 (14), 133 (14), 120 (6), 105 (12), 92 (100), 77 (10), 72 (7), 65 (24), 59 (92), 45 (25), 39 (7). HRMS (EI, 70 eV) calcd. for $\text{C}_{14}\text{H}_{22}\text{O}_4$ [M^+] 254, 1518. Found 254, 1515.

4.5. Diethylene glycol benzyl methyl ether (**21**)

A mixture of 0.1 mol diethylene glycol monomethyl ether, 0.11 mol benzyl chloride, 50% NaOH (60 mL), 0.005 mol tetrabutylammonium hydrogensulfate, and benzene (50 mL) was vigorously stirred at 70–80 °C on a water bath for 3 h. After cooling, water (100 mL) and hexane (100 mL) were added. The layers were separated, the organic layer was washed with water (3×50 mL) and dried with anhydrous MgSO_4 . Having removed hexane and benzene on a rotatory evaporator the residue was distilled under reduced pressure. Diethylene glycol benzyl methyl ether was finally obtained in 80%

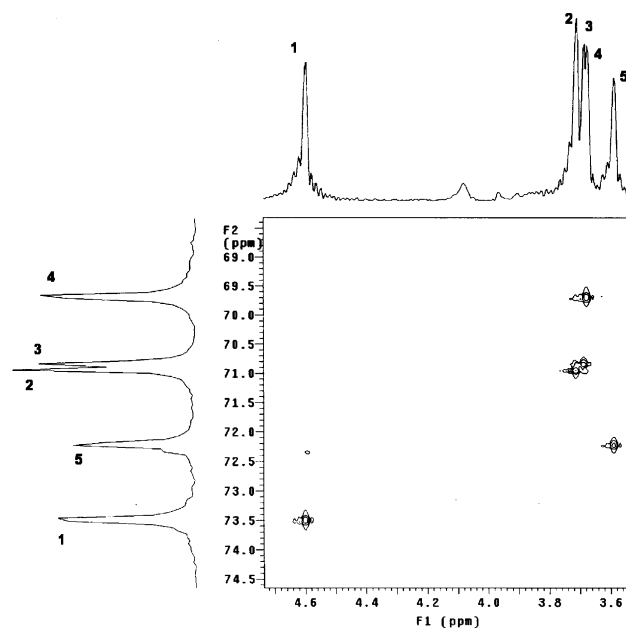
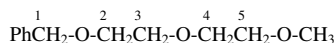


Fig. 1. 2D-NMR HETCOR (H, C) spectrum of diethylene glycol benzyl methyl ether (**21**) – methylene region.

yield as a novel compound (purity > 97%; GC).
B.p. = 163 °C/2 mm Hg.



¹H NMR (CDCl₃): 7.40–7.20 (m, 5H, Ph), 4.56 (s, 2H, CH₂-1), 3.60–3.65 (m, 6H, CH₂-2,3,4), 3.52–3.57 (m, 2H, CH₂-5), 3.37 (s, 3H, CH₃). ¹³C NMR: 138.13 (C-1, Ph), 128.21 (C_{meta}, Ph), 127.60 (C_{ortho}, Ph), 127.43 (C_{para}, Ph), 73.09 (CH₂-1), 71.81 (CH₂-5), 70.53 (CH₂-2), 70.42 (CH₂-3), 69.28 (CH₂-4), 58.90 (CH₃) (Fig. 1). MS, *m/z* (%): 210 (M⁺, 6), 134 (25), 107 (15), 91 (100), 76 (39), 59 (83), 45 (41). HRMS (EI, 70 eV) calcd. for C₁₂H₁₈O₃ [M⁺] 210, 1256. Found 210,1252.

4.6. Benzyl butyl ether (16), benzyl phenyl ether (6), and tetraethylene glycol benzyl vinyl ether (7)

Their GC–MS and NMR spectra have been reported in [6,7,14], respectively.

Acknowledgements

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