Formation and Stabilization of Free Radicals in Mixed-Crystal Structures of Dicarboxylic and Peroxydicarboxylic Acids

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Abstract: A new phenomenon is described—the formation of free radicals from peroxydicarboxylic acids (peroxydisuccinic and peroxydiglutaric) on their cocrystallization with dicarboxylic acids (succinic, glutaric, and fumaric acids) in the absence of any external action on the matrix. The free-radical species were detected by ESR. The steady-state concentration of the radicals depends on the molar ratio of peroxydicarboxylic acid/dicarboxylic acid, the maximum concentration being ca. 6 × 10^{18} radicals/g. Free radicals stabilized in dicarboxylic acid matrices could be stored as radical-containing samples for several months at ambient temperature. X-ray studies of the mixed peroxydicarboxylic acid–dicarboxylic acid crystals clearly point to the importance of the structural resemblance between the components as the main factor responsible for the formation of the free-radical species.

Matrix isolation is one of the most effective ways of free-radical fixation. Radical generation directly in the matrix is attained by the action of different types of radiation (radiolysis, photolysis, and electric discharge)[1] on the molecule trapped in it.

We have found a new phenomenon—the formation of free radicals from peroxydisuccinic and peroxydiglutaric acids (PDSA and PDGA) on their cocrystallization with dicarboxylic acids (succinic, glutaric, and fumaric acids: SA, GA, and FA, respectively) in the absence of any external action on the matrix.

Removal of the solvent on a Rotovap at 10–20 °C under 30–50 mmHg produced a crystalline residue, which was further analyzed by ESR to detect the presence of free-radical species. Control experiments have shown that pure crystalline PDSA and PDGA samples are quite stable and reveal no tendency to produce free radicals upon storage for several months at 0 °C. The same is true for the samples prepared by the mechanical mixing of crystalline PDSA and PDGA with SA, GA, and FA.

Other aliphatic dicarboxylic acids (C_6–C_{12}) fail to produce free-radical species upon their cocrystallization with PDSA or PDGA.

On the contrary, in the PDSA–SA samples prepared by the above cocrystallization procedure, ESR spectra revealed the presence of a signal (Figure 1a) whose structure is similar to that of 1,2-dicarboxyethyl radicals (I) observed earlier upon radiolysis of polycrystalline SA samples.[3] This signal is characterized by the following hyperfine splitting constants: a_α = 25 Oe, a_{β_1} = 39 Oe, and a_{β_2} = 25 Oe, which are close to those for radicals I observed in radiolyzed single crystals of pure SA[4] (a_α = 21.5 Oe, a_{β_1} = 39 Oe, and a_{β_2} = 29 Oe). The two middle components of the spectrum are less distinct, presumably owing to overlapping signals of other radicals present in the system under study.

The steady-state concentration of radicals I depends on the PDSA/SA = m molar ratio (Figure 2). The maximum concentration of I equal to 6 × 10^{18} radicals/g was observed at m = 0.025. The increase of the reagent molar ratio m leads to the concentration decrease of the formed radicals.

The concentration of the radicals does not depend on the surface size of the crystals. Thus, the PDSA–SA samples with the average crystal size 0.01 mm (from CH_3OH) and 1 mm (from acetone) contain comparable concentrations of radical species.

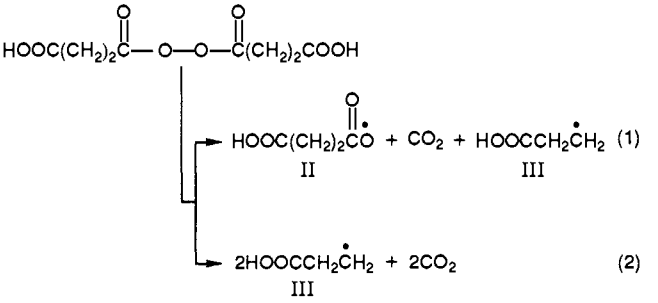
The formation of I is accompanied by decomposition PDSA and CO_2 evolution. The kinetics of the PDSA decomposition were determined in the solid-phase system PDSA–SA. The kinetic data were based on the measurement of the active oxygen concentration in the PDSA–SA solid-phase system (Figure 3).

As it is seen from the diagram, a fast peroxide decomposition is observed in the initial period, but later the reaction rate is considerably diminished. As follows from the data in Figure 3, the PDSA decomposition rates in the solid matrix increase by approximately 2 orders of magnitude when the temperature is increased from +6 to +45 °C.

PDSA decomposition rates in the solid SA matrix are considerably higher than those in liquid phase. For example, at 45 °C ~50% of the PDSA decomposition in the solid SA matrix occurs within ca. 1 h, while in acetic acid the same percentage of PDSA decomposition was reached only after ca. 3 h at 80 °C.[5]

These data, together with the known pattern of PDSA decomposition in solution, suggest the following mechanism of the free-radical formation in the solid matrix.

The initial stage is the O–O bond rupture in the PDSA molecule leading to the formation of radicals II and III:



Radicals II and III are formed directly in the SA matrix and cannot diffuse but participate in the H-atom abstraction from surrounding SA molecules, thus being transformed into more stable radicals I:

$$HOOC(CH_2)_2COOH + \dot{O}C(O)(CH_2)_2COOH \rightarrow HOOC\dot{C}HCH_2COOH + HOOC(CH_2)_2COOH \quad (3)$$

(II → I)

$$HOOC(CH_2)_2COOH + \dot{C}H_2CH_2COOH \rightarrow HOOC\dot{C}HCH_2COOH + CH_3CH_2COOH \quad (4)$$

The proposed mechanism is supported by the CO_2 evolution (ca. 1.4 mol/mol of PDSA) and by isolation of propionic acid (ca.

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Table I. Main Crystal Structure Parameters of Peroxydicarboxylic and Dicarboxylic Acids[a]

cell params	crystal structure				
	succinic acid (β form)[8]	fumaric acid (β form)[11]	glutaric acid (β form)[12]	peroxydisuccinic acid[7a]	peroxydiglutaric acid[10a]
a, Å	5.126 (10)	5.264 (3)	17.40 (5)	5.347 (1)	16.518 (7)
b, Å	8.88 (7)	8.974 (2) ($2b$)	4.87 (1)	9.019 (2)	4.308 (4) ($b/2$)
c, Å	7.619 (6)	7.618 (3)	15.28 (4)	7.092 (2) ($c/4$)	16.889 (8)
α, deg	90	106.85 (5)	90	102.04 (3)	90
β, deg	133.6 (1)	134.94 (8)	104.2 (4)	133.6 (3)	101.75 (4)
γ, deg	90	86.33 (5)	90	80.69 (3)	90

[a] Parameters of nonprimitive (specially transformed) unit cells.

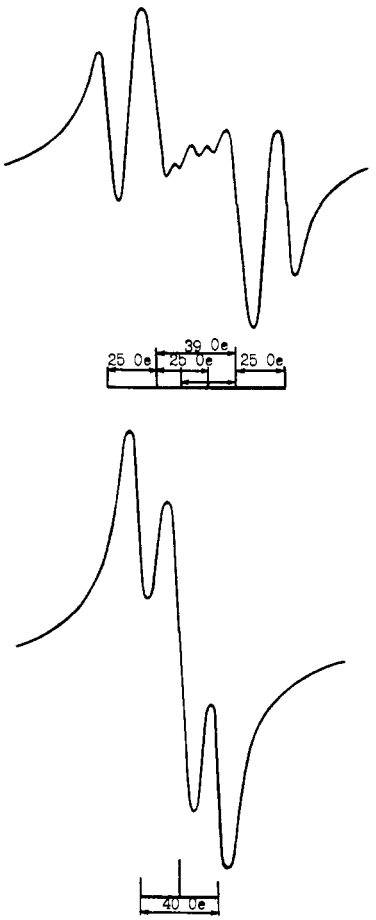

Figure 1. ESR spectra of radicals in crystalline systems: (top) peroxydisuccinic–succinic acids, (bottom) peroxydisuccinic–fumaric acids.

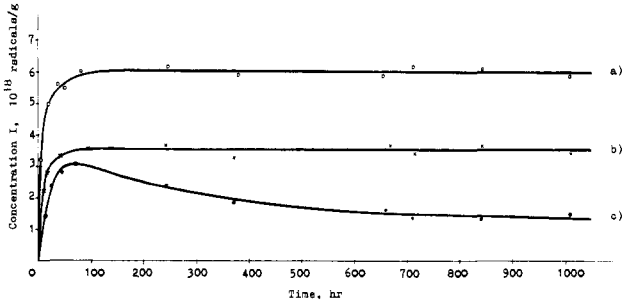

Figure 2. Kinetics of 1,2-dicarboxyethyl radicals (I) accumulation in the PDSA–SA crystalline system. Temperature, +20 °C. PDSA/SA molar ratio: (a) m = 0.025; (b) m = 0.05; (c) m = 0.1.

1.4 mol/mol of PDSA) from the PDSA–SA samples kept at 70 °C for 20 h.

The photolysis of pure crystalline samples of diacyl peroxide was accompanied by formation of the alkyl radicals and CO_2.[6]

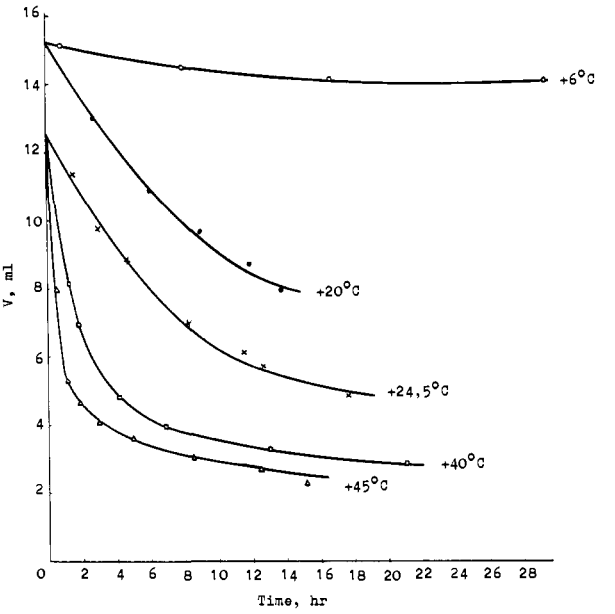

Figure 3. Kinetics of PDSA decomposition in the PDSA–SA crystalline system. PDSA-SA molar ratio m = 0.025. V, volume of $Na_2S_2O_3$ (0.1 N) required for titration of 1-g solid samples of PDSA–SA.

To get some insight into the nature of these solid-state interactions, X-ray analysis of a PDSA–SA single crystal was carried out for comparison with the relevant data for single-crystal analysis of PDSA[7] and SA.[8]

PDSA molecules are packed in the crystal similarly to those of SA (β form) as H-bonded chains. For the PDSA and SA structures an approximate isomorphism is observed (Table I), i.e., $a'_{PDSA} = 1.043a_{SA}$; $b'_{PDSA} = 1.016b_{SA}$; $c'_{PDSA}/4 = 0.931c_{SA}$. Since the length of the PDSA molecule in the H-bonded chain direction (14.19 Å) is less than the SA crystal cell dimension corresponding to two SA molecules (15.24 Å),[8] on inclusion into the SA matrix PDSA molecules should be subjected to longitudinal stretching forces. Hence the deformation of included PDSA molecules, which seems to consist mainly in the elongation of hydrogen bonds and the change of bond and torsion angles, may also lead to some elongation of the O–O bond, i.e., to its weakening.

Moreover, according to the crystal structural data, the nonplanar peroxide molecule has a larger effective cross section (17.04 Å^2) than two SA molecules (16.48 Å^2). Therefore, on inclusion of the PDSA molecule into the SA crystal lattice (Figure 4), a flattening of the peroxide groups and a trans-flattening of hydrocarbon chains can take place which, in its turn, should also lead to the elongation and weakening of the O–O bond.

An X-ray examination of samples obtained by the SA and PDSA cocrystallization (m = 0.01) was also carried out. (Practically stable crystals, which were kept under ambient temperature for 2 months after crystallization, were used.) These

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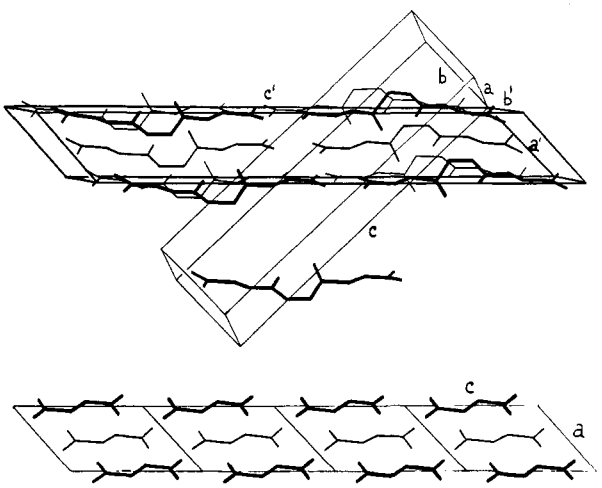

Figure 4. Projections of crystal structures of PDSA (top) and SA (bottom) onto the crystallographic planes $a'c'$ (PDSA) and ac (SA). The chains of molecules linked by H bonds run along the c' and c axes, respectively. Thin lines in the PDSA structure show the reduced primitive unit cell and thick lines show the transformed ($h' = -k$, $k' = 2h - k$, $l' = -h + 4k + l$)[7] C-centered unit cell illustrating the similarity of the structures of PDSA and SA (four unit cells in the SA structure are shown, $c_{PDSA} \approx 4c_{SA}$). The lines of different thickness are used to distinguish the molecules with different third coordinates (normal to the paper). The reference molecule in the PDSA structure is shown separately.

samples contained free 1,2-dicarboxyethyl radicals (recorded by ESR) and peroxydisuccinic acid. Besides "native" reflections (corresponding to the crystalline β form), the diffraction pattern also has reflections with fractional (relative to the "native" ones) values of indices. These reflections correspond to primitive monoclinic cells with doubled or tripled (in comparison with the SA β form) a, b, and c parameters and the same value of the β angle. Spatial orientations of all three crystal lattices coincide. Reflections with more than tripled parameters were not observed. However, it is obvious that reflections with "mixed" parameters of unit cells (for instance, a_{SA}, b_{SA}, $2c_{SA}$; $3a_{SA}$, b_{SA}, $3c_{SA}$; $2a_{SA}$, $3b_{SA}$, c_{SA}, etc., in all 24 possible combinations) may be superimposed onto the above-mentioned reflections.

Additional reflections of the "$(h,k,l)/2$" and "$(h,k,l)/3$" types are rather distinct. Although their experimental mean integral intensity is approximately 2 and 3 orders, respectively, lower than the intensity of the "native" reflections in the region of $\theta \leq 20°$, about 75 and 15% of these reflections have the intensity $I > 2\sigma$.

Unit cell linear parameters calculated from the accurately measured positions of the reflections with the indices $(h_{SA} + {}^1/_2)$, $(k_{SA} + {}^1/_2)$, $(l_{SA} + {}^1/_2)$ proved to be somewhat lower [a = 15.174 (2), b = 17.684 (5), c = 10.166 (1) Å; β = 133.52 (1)°] in comparison with the doubled parameter values calculated from the "native" reflections [$2a_{SA}$ = 15.225 (1), $2b_{SA}$ = 17.737 (2), $2c_{SA}$ = 10.201 (1) Å; β_{SA} = 133.53 (1)°]. The corresponding cell volumes are equal to V = 1978.0 (7) Å^3 and $8V_{SA}$ = 1997.1 (3) Å^3, i.e., on doubling of the cell parameters the volume decrease calculated for one SA molecule amounts to 1.2 Å^3 only. Assuming the packing density constant and using atomic and group volume increments,[9] this volume change can be assigned to the loss of either one H atom by every second SA molecule or a carboxyl group (in the form of CO_2) by every sixteenth molecule.

The observed effect resulting from the presence of "doubled" and "tripled" lattices, alongside of the "native" lattice, may be satisfactorily intepreted by the existence of domain regions in the samples under study. These regions represent stoichiometric highly ordered solid solutions of PDSA or free radicals I in the SA matrix.

Similar processes of 1,3-carboxy-1-propyl radical IV generation and stabilization are observed on PDGA cocrystallization. An ESR quadruplet is similar to that ascribed to radical I. Hence the formation of free radicals in the PDGA–GA system proceeds in a manner similar to that described above for the PDSA–SA system. However, the concentration of radical IV, formed in the PDGA–GA system, is much lower ($\sim 10^{17}$ radicals/g) than that of radical I, formed in the PDSA–SA system.

Most probably a lower concentration of radical IV is due to the lesser similarity of geometric parameters of the PDGA–GA crystalline system[11] in comparison with the PDSA–SA system.

Thus, with approximately equal contractions of the transition along the H-bonded chains in the PDGA structure relative to GA and in the PDSA structure relative to SA ($a_{PDGA} = 0.949a_{GA}$ and $c_{PDSA}/4 = 0.931c_{SA}$), the difference of two other parameters (b and c) in PDGA and GA amounts to ca. 10%. The latter value is significantly greater than that in the case of PDSA and SA (not more than 4%; see Table I). Thus, the inclusion of isolated molecules of PDGA into the GA crystal lattice is less favorable and less radical IV is formed.

For the PDSA–FA solid system the ESR data also indicated the formation of free radicals. However the ESR spectral parameters of these species were clearly different from those observed for I and IV. In the samples obtained by the cocrystallization of PDSA and FA (1:10 molar ratio) a triplet signal with a hyperfine splitting constant of ca. 20 Oe was detected by ESR (Figure 1b).[14]

We ascribed the structure V to the radicals detected in the PDSA–FA system and assume that in this case the solid-state homolysis of PDSA is accompanied by the addition of the initially formed radicals to the double bond of the FA matrix:

$$HOOCCH{=}CHCOOH + \dot{R} \rightarrow HOOCCHR\dot{C}HCOOH \quad (5)$$

where

$$R = HOOCCH_2\dot{C}H_2,\ HOOCCH_2CH_2C\dot{O}O \text{ radicals}$$

The formation of radicals in the PDSA–FA system is especially noteworthy in light of the complete absence of ESR signals in the mixed PDSA–MA crystals. This difference could be taken as evidence of the importance of the geometrical similarities of the cocrystallized substrates. In fact PDSA and FA are approximately isomorphous, while PDSA and MA reveal sharply different crystal structures (Table I).

The stability of alkyl radicals I, IV, and V in dicarboxylic acid matrices is especially noteworthy. Thus, ESR data indicated that on average less then 10% of the initial radical I content is lost upon the storage of the samples for 1 month in open air at ambient temperature. These results imply that the rate of radical disproportionation and/or recombination as well as the rate of their interaction with oxygen is greatly reduced upon the inclusion of radical species in solid matrices of dicarboxylic acids.

Experimental Section

The ESR spectra were recorded with an X-band spectrometer. Unit cell parameters and X-ray reflection intensities of peroxydicarboxlic acids were measured at −120° with an automated four-circle Syntex $P2_1$ diffractometer. The PDGA and PDSA structures were solved by the direct method (MULTAN program) and refined by full-matrix least squares in anisotropic approximation for non-hydrogen atoms to R = 0.049 (PDSA) and 0.074 (PDGA).[7,10] A diffraction pattern of mixed SA and PDSA crystals was recorded with a Hilger-Watts Y290 diffractometer at 20°.

Peroxydicarboxylic acids (PDSA and PDGA) were prepared from acid anhydrides and H_2O_2.[14] Mixed-crystal samples were prepared by dissolving the peroxides (PDSA or PDGA) and respective dicarboxylic acids (SA, FA, GA, MA) in methanol or acetone followed by the complete evaporation of the solvent on a Rotavap (20 °C, 20 mmHg).

For the ESR spectra measurements the crystalline samples were put into ampules and vacuum pumped to the residual pressure of about 10^{-3}–10^{-4} Torr.

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The ESR spectra were recorded with an X-band spectrometer (modulation frequency of 1 Hz). Radical concentrations were measured with a single-crystal $CuCl_2 \cdot 2H_2O$ as a reference sample.[15]

The CO_2 yield were determined according to the published procedure.[16] The sealed tube with a sample under study (~0.5 g) was kept thermostated at a preset temperature, and then it was attached to a vacuum system and the seal was broken. Methanol vapor was condensed at -196 °C (~2.0 mL) and the sample was melted. The homogeneous solution produced was cooled to -80 °C and CO_2 and methanol were recondensed from the gas phase into an intermediate trap at -196 °C. The trap with condensate was warmed to -80 °C and CO_2 was again condensed in a V-shaped trap at -196 °C. The latter was warmed to -80 °C and the CO_2 pressure was measured with an oil manometer. These operations were repeated several times until a constant CO_2 pressure was attained. The accuracy of the measurement was ca. 10%.

Kinetic measurements of the PDSA decomposition rate in the PDSA-SA solid-phase system were made in a thermostatic reactor in an Ar atmosphere, where the crystalline samples prepared according to the above procedure were placed. At indicated time intervals the solid samples were removed and peroxide concentration was determined by the iodometric method.[17]

Propionic acid was identified in the PDSA-SA decomposition products by GLC. The initial sample (3.02 g) with 0.22-g PDSA content was placed into a round-bottom flask, which was preheated in a thermostat at 70 °C until the ESR signal entirely disappeared (~200 h). Reaction products were extracted with *n*-hexane and analyzed by GLC, using an internal standard (*n*-valeric acid). The propionic acid content in the reaction products was ~0.101 g. Analysis conditions: a 3 mm × 2 m column with SE-30 [5% on a Chromosorb W (60-80 mesh)]; temperature, 110 °C; gas carrier, nitrogen.

Registry No. I, 5905-59-9; IV, 16405-28-0; $HO_2CCH_2\dot{C}H_2$, 2887-43-6; $HO_2C(CH_2)_2COO\cdot$, 6233-24-5; $HO_2CCH((CH_2)_2CO_2H)\dot{C}HCO_2H$, 123290-22-2; $HO_2CCH(O_2C(CH_2)_2CO_2H)CHCO_2H$, 123290-23-3; succinic acid, 110-15-6; fumaric acid, 110-17-8; glutaric acid, 110-94-1; peroxydisuccinic acid, 123-23-9; peroxydiglutaric acid, 10195-54-7.

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Crown Ether Alcohols. 1. Crystal and Molecular Structure of the Complex between *sym*-Hydroxydibenzo-14-crown-4 and Water Molecules ($[C_{18}H_{20}O_5] \cdot 1.25[H_2O] \cdot 0.125[CH_3OH]$) Including Interesting Water-Methanol Channels

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Contribution from the Department of Chemical Services, Weizmann Institute of Science, Rehovot 76100, Israel, the Department of Chemistry and Biochemistry, Texas Tech University, Lubbock, Texas 79409, and the Department of Analytical and Inorganic Chemistry, The Hebrew University of Jerusalem, Jerusalem 91904, Israel. *Received December 21, 1988. Revised Manuscript Received July 21, 1989*

Abstract: The synthesis and crystal structure of the title compound are described. Single-crystal X-ray structure analysis indicates 8 formula units in the unit cell of parameters $a = 16.024$ (1) Å and $c = 13.076$ (1) Å. The space group is $I\bar{4}$. Direct methods yielded the structure, which was refined by least-squares techniques to a final R factor of 0.038 for 1533 independent observations. A water molecule forms a 1:1 neutral complex with the crown ether alcohol. In this complex, the water molecule is hydrogen bonded to the crown ether hydroxyl group. Four monohydrate complexes are hydrogen bonded to a central water molecule in a perfect tetrahedral geometry and to an apical methanol molecule. Unusual water-methanol channels are found in this structure. The crystal packing of the complex includes hydrophilic water-methanol channels which are surrounded by hydrophobic cylinders consisting mainly of benzo rings and methylene groups. The crystal structure provides a model for the encapsulation of water molecules by hydrophobic regions with potential application for the formation of hydrophilic pores in biological bilayers.

Macrocylic polyethers (crown ethers) are known to form stable and often selective complexes with various cations.[1,2] Recently, these polyethers have been found to form complexes with neutral molecules as well.[3] Among the class of charged complexes the guests include protons and hydronium ions, and among the neutral guests are water molecules. Relatively little information is available concerning complexes of protons[4-7] and/or hydronium ions[8-14] with cyclic polyethers and other ionophores.[15-17] It is not surprising, therefore, that the interactions of water molecules with neutral ionophores, which are essential for the formation of the ionophore-hydronium ion complexes, have not yet received appropriate attention.[16-34]

The binding between crown ethers and protons in aprotic organic solvents is primarily due to interaction of the protons with

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