4

Hydrogen Atom and Hydrated Electron

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4.1 Some Basic Properties of H• and e_{aq}^-

Much of basic free-radical chemistry of DNA and its constituents have been elucidated with the help of radiation techniques. This requires one to address briefly the properties of the H atom and the hydrated electron, e_{aq}^-, which are important intermediates in the radiolysis of water (Chap. 2.2).

The •OH radical, which is also generated under these conditions, may be converted into •H by reacting it with excess H_2 (Christensen and Sehested 1983). This may require a special pressure cell (Christensen and Sehested 1980). The scavenging of •OH with *t*BuOH ($k = 6 \times 10^8$ dm^3 mol^{-1} s^{-1}) is often the more convenient approach. Under adequate conditions, this leaves H• largely untouched, since its rate of reaction with *t*BuOH is low ($k = 1.7 \times 10^5$ dm^3 mol^{-1} s^{-1}; Buxton et al. 1988; recently revised at 1.15×10^6 dm^3 mol^{-1} s^{-1}; Wojnárovits et al. 2004).

H• is the conjugate acid of e_{aq}^- [pK_a(H•) = 9.1; reactions (1), $k = 2.2 \times 10^7$ dm^3 mol^{-1} s^{-1} and (2), $k = 2.3 \times 10^{10}$ dm^3 mol^{-1} s^{-1} (Buxton et al. 1988), for the thermodynamic properties of this system, see Hickel and Sehested (1985)]. Thus, in pure water, the lifetime of e_{aq}^- is quite long (Hart et al. 1966), even long enough to monitor its presence spectrophotometrically under steady-state ^{60}Co-γ-radiolysis conditions (Gordon and Hart 1964).

$$H^\bullet + OH^- \rightleftharpoons e_{aq}^- + H_2O \tag{1}$$

$$e_{aq}^- + H^+ \rightarrow H^\bullet \tag{2}$$

Reaction (1) is best described as a proton transfer from the weak acid H• to the strong base OH^- (Han and Bartels 1992). For the rapid conversion of e_{aq}^- into H• in neutral solution (i.e., at low H^+ concentration), phosphate buffer may be used [reaction (3); $k = 1.1 \times 10^7$ dm^3 mol^{-1} s^{-1} (Grabner et al. 1973)]. The rate constant depends somewhat on the phosphate concentration, and at 1 mol dm^{-3} phosphate (pH ~5.7) the reported value is 1.85×10^7 dm^3 mol^{-1} s^{-1} (Ye and Schuler 1986).

$$e_{aq}^- + H_2PO_4^- \rightarrow H^\bullet + HPO_4^{2-} \tag{3}$$

The hydrated electron is characterized by its strong absorption at 720 nm (ε = 1.9×10^4 dm^3 mol^{-1} cm^{-1} (Hug 1981); the majority of the oscillator strength is derived from optical transitions from the equilibrated *s* state to the *p*-like excited state (cf. Kimura et al. 1994; Assel et al. 2000). The 720-nm absorption is used for the determination of its reaction rate constants by pulse radiolysis (for the dynamics of solvation see, e.g., Silva et al. 1998; for its energetics see, e.g., Zhan et al. 2003). H• only absorbs in the UV (Hug 1981), and rate constants have largely been determined by EPR (Neta et al. 1971; Neta and Schuler 1972; Mezyk and Bartels 1995) and competition techniques (for a compilation, see Buxton et al. 1988). In many aspects, H• and e_{aq}^- behave very similarly, which made their distinction and the identification of e_{aq}^- difficult (for early reviews, see Hart 1964; Eiben 1970; Hart and Anbar 1970), and final proof of the existence of the

latter was only obtained with the report of its EPR spectrum in frozen alkaline solution (Schulte-Frohlinde and Eiben 1962; Eiben and Schulte-Frohlinde 1965) and the advent of pulse radiolysis (Boag and Hart 1963; Keene 1963, 1964). There is now vast literature on the physical properties and reaction kinetics of e_{aq}^-, and the latter data are only paralleled in number by those of $^{\bullet}OH$ (Buxton et al. 1988).

4.2 Redox Reactions

The hydrated electron is the most powerful reductant ($E_7 = -2.9$ V); $H^{\bullet}$ has a somewhat higher reduction potential ($E_7 = -2.4$ V; for a compilation of reduction potentials, see Wardman 1989). Often, both $^{\bullet}H$ and e_{aq}^- are capable of reducing transition metal ions to their lower oxidation states [e.g., reactions (4) and (5)].

$$Ag^+ + H^{\bullet} \rightarrow Ag + H^+ \tag{4}$$

$$Ag^+ + e_{aq}^- \rightarrow Ag \tag{5}$$

However, there are cases where the reduction potential of $H^{\bullet}$ is insufficient to reduce the metal ion, and the reduction reaction is only given by e_{aq}^- [e.g., reaction (6) (Baxendale and Dixon 1963); for a review see Buxton and Sellers (1977); for a compilation of rate constants of ensuing reactions see Buxton et al. (1995)].

$$Zn^{2+} + e_{aq}^- \rightarrow Zn^+ \tag{6}$$

In strongly acid solution, $H^{\bullet}$ may even react as an oxidant. For example, $H^{\bullet}$ oxidizes Fe^{2+} to Fe^{3+} [reaction (7)]. A hydride, $Fe^{3+}H^-$, is thought to be an intermediate in this reaction.

$$Fe^{2+} + H^{\bullet} + H^+ \rightarrow Fe^{3+} + H_2$$

4.3 Dissociative Electron Capture and Related Reactions

The hydrated electron reacts with many compounds which are capable of releasing an anion by dissociative electron capture [e.g., reaction (8)], and, among others, it was this property which allowed the differentiation between e_{aq}^- and $H^{\bullet}$ [reactions (9) and (10)] (Armstrong et al. 1958; Hayon and Allen 1961; Jortner and Rabani 1962).

$$e_{aq}^- + ClCH_2CO_2H \rightarrow Cl^- + {}^{\bullet}CH_2CO_2H \tag{8}$$

$$H^{\bullet} + ClCH_2CO_2H \rightarrow H_2 + {}^{\bullet}CHClCO_2H \tag{9}$$

The analogous reactions with N_2O (Dainton and Peterson 1962) are commonly used to convert e_{aq}^- into $^\bullet OH$ [reaction (10); $k = 9.1 \times 10^9$ dm^3 mol^{-1} s^{-1} (Janata and Schuler 1982)], since N_2O is largely inert against free-radical attack. Some much slower reactions, often leading to chain reactions, are given by other strongly reducing radicals (Cheek and Swinnerton 1964; Sherman 1967a-c; Ryan and Freeman 1977; Ryan et al. 1978; Al-Sheikhly et al. 1985; Wang et al. 1996).

$$e_{aq}^- + N_2O \rightarrow N_2 + O^{\bullet -} \quad (O^{\bullet -} + H_2O \rightarrow {}^\bullet OH + OH^-) \tag{10}$$

The alternative of using H_2O_2 [reaction (11); $k = 1.3 \times 10^{10}$ dm^3 mol^{-1} s^{-1}] instead of N_2O may lead to complications, since H_2O_2 also reacts with $^\bullet OH$, $^\bullet H$ and many other radicals (Chap. 6).

$$e_{aq}^- + H_2O_2 \rightarrow {}^\bullet OH + OH^- \tag{11}$$

In their reactions with tertiary butylhydroperoxide, $^\bullet H$ and e_{aq}^- show a different selectivity (Phulkar et al. 1990). While $^\bullet H$ undergoes reactions (12) and (13) with about equal probability, i.e., both tBuO$^\bullet$ and $^\bullet OH$ are formed, e_{aq}^- yields only tBuO$^\bullet$ [reaction (14)]. This preference in splitting the peroxidic bond is due to the much higher solvation energy of the hydroxide compared to the tertiary butoxide ion. For a detailed study on the reaction of $^\bullet H$ with H_2O_2 see Mezyk and Bartels (1995).

$$t\text{BuOOH} + H^\bullet \rightarrow t\text{BuO}^\bullet + H_2O \tag{12}$$

$$t\text{BuOOH} + H^\bullet \rightarrow t\text{BuOH} + {}^\bullet OH \tag{13}$$

$$t\text{BuOOH} + e_{aq}^- \rightarrow t\text{BuO}^\bullet + OH^- \tag{14}$$

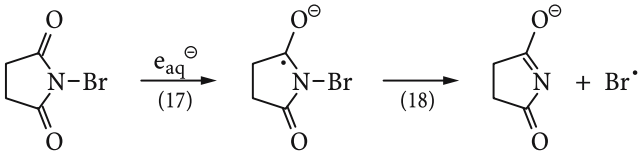
With thiols, e_{aq}^- reacts predominantly by dissociative ET [reaction (15)], but also formation of molecular hydrogen [reaction (16)] is observed (Hoffman and Hayon 1973). As one might expect, there is a considerable drop in the rate constant when the thiol group is deprotonated (for a systematic study, see Mezyk 1995).

$$e_{aq}^- + RSH \rightarrow R^\bullet + SH^- \tag{15}$$

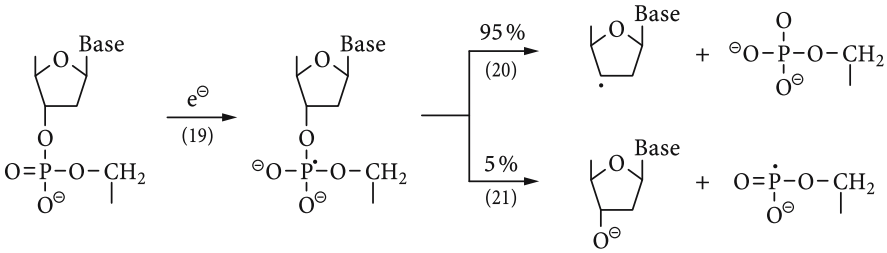
$$e_{aq}^- + RSH \ (+ H^+) \rightarrow RS^\bullet + H_2 \tag{16}$$

In a number of reactions that are written as dissociative electron attachments, short-lived radical anions are in fact intermediates. A case in point is 5BrUra (Chap. 10). An interesting behavior is shown by the radical anion of *N*-bromosuccinimide which does not release a bromide ion but rather fragments into a bromine atom and a succinimide anion [reactions (17) and (18)] (Lind et al. 1991).

The concerted (one-step) dissociative ET is undergone by σ^* electrophores; the $(\sigma^* + \pi^*)$ electrophores give rise to two-step processes (Savéant 1993; Schmittel and Ghorai 2001).



The DNA backbone could be split, in principle, by dissociative electron capture [reactions (19)-(21)].



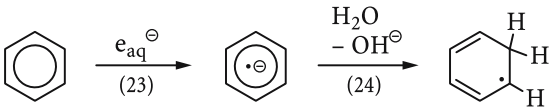
DNA strand breakage by e_{aq}^- is not observed in dilute aqueous solution (von Sonntag 1987). DFT calculations show that these reactions are exoenergetic but require activation energy (Li et al. 2003; for quantum-mechanical calculations, see Berdys et al. 2004a; for base radical anions serving as a relay, see Berdys et al. 2004b). It has been envisaged that low-energy-electrons could induce this reaction in competition with other reactions such as thermalization/solvation and addition to the bases (for reviews on the reactions of low-energy electrons in their reactions with DNA, see Sanche 2002a,b). Reactions (19)-(21) have been indeed observed under certain conditions (Becker et al. 2003; Chap. 12). Subexcitation electrons (<3 eV) may also release Thy from DNA (Abdoul-Carine et al. 2004).

4.4 Addition Reactions

Dissociative electron capture can only occur when single-bonds are involved. There are, however, a large number of compounds with high electron affinity such as O_2 and carbonyl, nitro- or cyano-groups containing compounds. With all these compounds, e_{aq}^- reacts at diffusion-controlled rates by forming the corresponding radical anion [e.g., reactions (22); $k = 1.9 \times 10^{10}$ dm^3 mol^{-1} s^{-1}]. The radical anions thus-formed are themselves usually strong reductants (including, for example, the Thy radical anion; Chap. 10), and readily hand over an electron to a better electron acceptor (for ketyl radical anions see, e.g., Adams and Willson 1973; for nucleobase radical anions see, e.g., Adams and Willson 1972; Nese et al. 1992; for $O_2^{\bullet-}$ see Chap. 8).

$$e_{aq}^- + O_2 \rightarrow O_2^{\bullet-} \qquad (22)$$

Simple olefins do not react with e_{aq}^- at an appreciable rate, but compounds with an extended π-system such as butadiene can also accommodate an additional electron ($k = 8 \times 10^9$ dm^3 mol^{-1} s^{-1}; Hart et al. 1964). However, as in the case of benzene, the rate is often below diffusion controlled [reaction (23); $k = 7.2 \times 10^6$ dm^3 mol;^{1} s;^{1} (Gordon et al. 1977); in THF, the reaction of the solvated electron with benzene is even reversible (Marasas et al. 2003)], and the resulting radical anion is rapidly protonated by water [reaction (24)].



A rapid protonation by water of the electron adducts of spin traps such as DMPO or 2-methyl-2-nitroso-propane yields the same species as are expected for the reaction of H• (Sargent and Gardy 1975). This prevents a distinction between e_{aq}^- and H• by using this technique.

H• readily adds to C–C double bonds. Like •OH, it is a pronounced electrophilic radical (ρ = −0.45; Neta 1972) and thus shows a high regioselectivity in its addition reactions. With e_{aq}^-, it shares a fast reaction with O_2 [reaction (25); $k = 1.2 \times 10^{10}$ dm^3 mol^{-1} s^{-1}].

$$H^{\bullet} + O_2 \rightarrow HO_2^{\bullet} \tag{25}$$

4.5 H-Abstraction Reactions

H• also undergoes H-abstraction reactions, albeit with much lower rates than •OH. This is also reflected in a higher H/D isotope effect [e.g., with 2-PrOH/2-PrOH-d_2 $k_H/k_D \approx 7.5$, reactions (26) and (27) (Anbar and Meyerstein 1964); see also Vacek and von Sonntag (1969), vs. $k_H/k_D = 1.5$ for •OH (Anbar et al. 1966)].

$$^{\bullet}H + HC(CH_3)_2OH \rightarrow H_2 + {}^{\bullet}C(CH_3)_2OH \tag{26}$$

$$^{\bullet}H + DC(CH_3)_2OH \rightarrow HD + {}^{\bullet}C(CH_3)_2OH \tag{27}$$

If there is competition between addition and H-abstraction, addition is always preferred. As a consequence, H-abstraction from the sugar moiety is a very minor process in DNA and related compounds (Das et al. 1985).

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