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Spectroscopic study of selenadiazoloquinolones in alkaline media

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Abstract

Newly synthesized derivatives of 6–oxo–6,9–dihydro[1,2,5] selenadiazolo [3,4–h] quinoline variously substituted at position 7 are witnessed in solution in the N_9–deprotonated and protonated oxo tautomeric forms depending on the pH using UV/vis and NMR spectroscopy. Upon the anodic oxidation selenadiazoloquinolones produce paramagnetic species observed by EPR spectroscopy.

Keywords: UV/vis spectroscopy, NMR spectroscopy, selenadiazoloquinolones.

Introduction

4–Oxoquinolines represent a group of heterocyclic compounds widely applied in the medical care. The presence of selenium in molecules can indicate new effects, and some of synthesized selenaheterocyclic compounds demonstrated attractive biological impact. Novel 7–substituted 6–oxo–6,9–dihydro[1,2,5] selenadiazolo[3,4–*h*]quinoline (R = H, $COOC_2H_5$, $COOCH_3$, COOH, $COCH_3$ and CN) were synthesized as potential anticancer and antimicrobial agents. The presented work deals with the formation of radical species from selenadiazoloquinoline derivatives generated upon electrochemical oxidation in alkaline media at various pH values and monitored by *in situ* EPR spectroscopy.

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Materials and methods

The electrochemical EPR experiments were performed in the alkaline aqueous solutions (0.1 M or 0.001 M NaOH) saturated with selena–diazoloquinolones. ^1H and ^{13}C NMR spectra were recorded with Varian VNMRS 600 MHz spectrometer equipped with the ^{13}C Enhanced Salt Tolerant Triple Resonance coldprobe in 0.1M NaOH/10% D_2O solution at 25°C and acetone as reference standard ($^1H/^{13}C$ methyl 2.22/30.89 ppm). The UV/visible spectra of investigated selenadiazoloquinolones in aqueous solutions at different pH were recorded using a UV–3600 UV/Vis spectrometer (Shimadzu, Japan).

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Results and Discussion

UV/vis experiments showed that two forms of selenadiazoloquinolones are present in the solution, depending on the pH. As the observed behavior cannot be explained by the

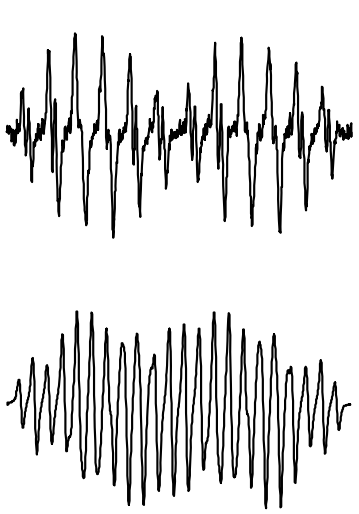

Figure 1: EPR spectra observed by the anodic oxidation of 6–oxo–6,9–dihydro[1,2,5] selenadiazolo[3,4–*h*]quinoline in aqueous (a) 0.1 and (b) 0.001 NaOH (magnetic field sweep width 1.0 mT).

tautomeric hydroxy/oxo exchange (as evidenced by the NMR data) the two forms can only result from the deprotonation/protonation of

the acidic hydrogen at N9 of the 4–pyridone moiety. The EPR spectra measured upon the anodic oxidation of unsubstituted derivative in 0.1 and 0.001 NaOH originate from the N9–deprotonated and protonated oxo–tautomeric forms, respectively (Fig 1). However, their simulation analysis revealed no hyperfine splittings corresponding to N1, N3 and 77Se nuclei. Consequently, we assume that absence of primary paramagnetic oxidation products is caused by the alkaline conditions coupled with high polarization potential (~1.5 V), and the initial oxidation step is followed by hydrolysis of selenadiazolo moiety producing ortho-semiquinone paramagnetic species.

Conclusion

Under alkaline conditions investigated selenadiazoloquinolones exist in the oxo–tautomeric forms. EPR *in situ* electrochemical oxidation revealed the formation of paramagnetic species most probably characterized with semiquinone structure.

Acknowledgement

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