

Conclusions

Thus, the results clearly demonstrated that mixing CN with CA and the strategic use of additives can lead to the successful development of highly efficient CN-based membranes for UF separation of proteins. This makes them promising for use for separation and purification in the pharmaceutical industry.

Acknowledgements

The study was supported by the Russian Science Foundation grant No. 20-79-10064, <https://rscf.ru/en/project/20-79-10064/>. The experimental work was facilitated by equipment from the Resource Centers for Nanotechnology, Magnetic Resonance, Cryogenic Department, Thermogravimetric and Calorimetric Research Center; Center for Physical Methods of Surface Investigation, Center for Innovative Technologies of Composite Nanomaterials, Chemical Analysis and Materials Research Center, and Center “Nanofabrication of Photoactive Materials (Nanophotonics)” at the St. Petersburg State University.

Determination of the structure of regioisomeric furan-containing naphthofuranes based on 1D and 2D NMR spectroscopy experiments

Minaeva V. Yu., Ozerova O. Yu., Makarenko S. V.

Herzen State Pedagogical University of Russia, Department of Organic Chemistry

48 Moyka River Embankment, Saint Petersburg 191186, Russia

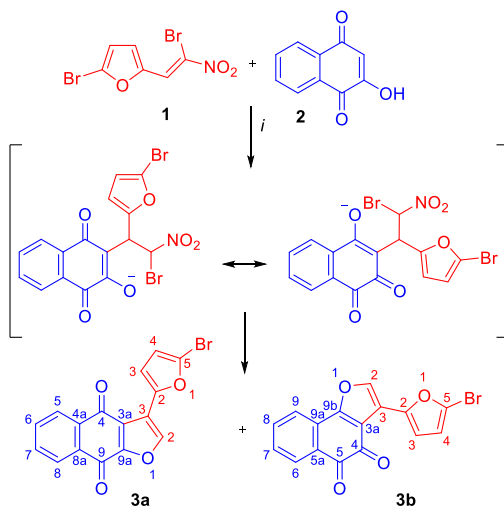
E-mail: kohrgpu@yandex.ru

http://kohrgpu.ru

We have previously shown that the interaction of furan-containing *gem*-bromonitroethenes with representatives of cyclic CH-acids leads to the formation of tri- and tetracyclic heterocyclic structures of the dihydrofuran and furan series [1].

The study of the reaction of a representative of furan-containing *gem*-bromonitroethenes – 2-(5-bromofuryl)-1-bromo-1-nitroethene **1** with 2-hydroxynaphthoquinone-1,4 **2** showed that the process leads to the formation of a mixture of regioisomeric 3-(5-bromofuran-2-yl)naphtho[2,3-*b*]furan-4,9-dione **3a** and 3-(5-bromofuran-2-yl)naphtho[1,2-*b*]furan-4,5-dione **3b** (ratio 1:1.17 according to the ^1H NMR spectrum), which was successfully separated into individual isomers by recrystallization (Scheme 1).

Scheme 1.



i: C_6H_6 , Et_3N , 3 h, reflux or MeOH, AcOK, 3d, r.t.

The synthesized 3-(5-bromofuran-2-yl)naphtho[2,3-*b*]furan-4,9-dione **3a** and 3-(5-bromofuran-2-yl)naphtho[1,2-*b*]furan-4,5-dione **3b** can exist as *s-cis*- and *s-trans*-conformers (Fig. 1). The presence of one set of signals in the ^1H and ^{13}C NMR spectra of individual substances **3a**, **b** indicates that they are formed by configurationally homogeneous (Fig. 1).

Thus, the aim of this work is to determine the fine structure of 3-(5-bromofuran-2-yl)naphtho[2,3-*b*]furan-4,9-dione **3a** and 3-(5-bromofuran-2-yl)naphtho[1,2-*b*]furan-4,5-dione **3b** by 1D and 2D NMR spectroscopy experiments.

Two doublets at 6.46 and 7.63 ppm ($^3J = 3.5$ Hz) of the C³H and C⁴H protons of the furan ring, a singlet at 8.14 ppm of the C²H proton and two multiplets of the protons of the condensed benzene ring in the regions 7.86–7.75 ppm и 8.27–8.19 ppm are observed in the ¹H NMR spectrum of isomer 3a (Fig. 2).

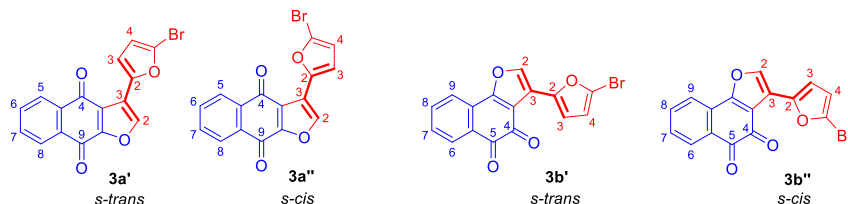


Figure 1. Possible conformational isomers of compounds 3a and 3b.

In the ¹H-¹³C HMBC spectrum of isomer 3a, cross peaks of the downfield multiplet of the benzene ring in the region of 8.27–8.19 ppm and signals of carbon atoms at 173.63 and 180.20 ppm are observed, which is possibly a consequence of the interaction of the C⁵H and C⁸H protons with the carbonyl carbon atoms C⁴ and C⁹, confirming the structure of the regioisomer 3a (Fig. 3).

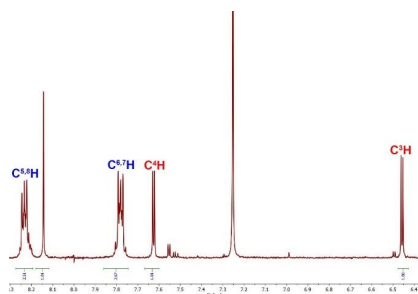


Figure 2. ¹H NMR spectrum of the compound 3a (CDCl₃).

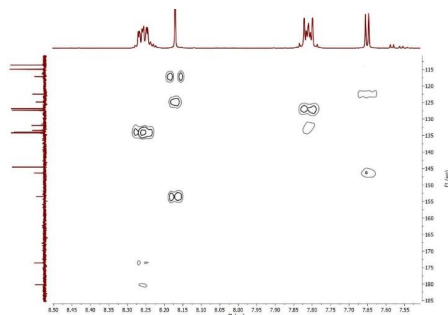


Figure 3. ¹H-¹³C HMBC NMR spectrum of the compound 3a (CDCl₃).

The study of compound 3a by the ¹H-¹H NOESY experiment showed that the C³H and C⁴H protons of the furan ring, as well as C⁵H, C⁶H, C⁷H, C⁸H, regularly demonstrate the Nuclear Overhauser Effect, while the C²H proton does not correlate with the C³H and C⁴H protons of the furan ring, which allows us to assign the *s-trans* configuration to this compound (Fig. 4).

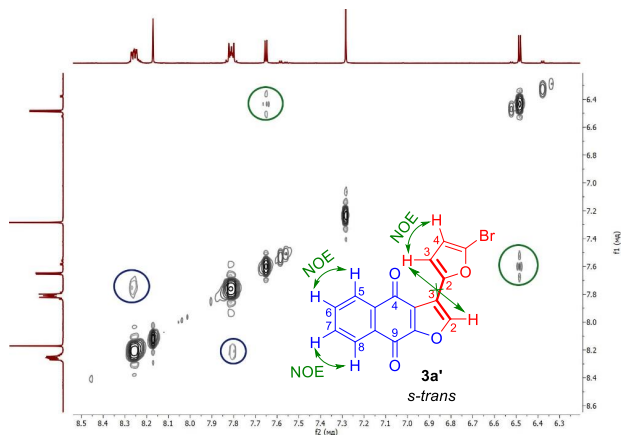


Figure 4. ^1H - ^1H NOESY spectrum of the compound **3a** (CDCl_3)

Thus, based on the results of the ^1H - ^1H NOESY experiment, the *s-trans* configuration of the obtained 3-(5-bromofuran-2-yl)naphtho[2,3-*b*]furan-4,9-dione **3a** was established. The configuration of 3-(5-bromofuran-2-yl)naphtho[1,2-*b*]furan-4,5-dione **3b** is undergoing further studies.

References

1. Minaeva V.Yu., Marshev E.D., Popov G.S., Pilipenko I.A., Stepanova A.M., Ozerova O.Yu., Makarenko S.V. Synthesis of tri- and tetracyclic bifuran structures based on furan-containing gem-bromonitroethenes // Abstracts of the II Interdisciplinary All-Russian youth scientific school-conference with international participation "Molecular design of biologically active substances: biochemical and medical aspects". Kazan. September 16-20 2024. P. 115.