ISSN 1070-3632, Russian Journal of General Chemistry, 2009, Vol. 79, No. 6, pp. 1183–1190. © Pleiades Publishing, Ltd., 2009.
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Thermal Behavior of Quinoline *N*-Oxide Hydrates and Deuterohydrate

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Received December 22, 2008

Abstract—Spectral and thermochemical investigation of physicochemical properties of quinoline *N*-oxide crystallohydrates with H_2O and D_2O is carried out. Quinoline *N*-oxide is established to form with H_2O a stable dihydrate where two water molecules are energetically not equal. Complete dehydration of quinoline *N*-oxide occurs when temperature reaches 150°C. With accounting for the obtained thermochenical data, quinoline *N*-oxide and its mono- and dihydrates are isolated in the individual state and their IR spectra are registered and considered. It is established that at boiling quinoline *N*-oxide in D_2O proceeds chemical reaction affording isoindoline-1,3-dione (phthalimide). The product is identified by elemental analysis and ^1H NMR and IR spectroscopy. The band assignment in the IR spectra of quinoline *N*-oxide, phthalimide and of the complex of the latter with D_2O is based on the quantum-chemical DFT calculations.

DOI: 10.1134/S1070363209060267

Due to structural features of heterocyclic *N*-oxides (high donor properties of N → O group and its steric accessibility) these compounds are important synthones and pharmaceutic substances. Due to high reactivity of oxygen atom in the *N*-oxide molecule they are active toward proton-donor molecules. For example, in [1] has been shown that at the protonation of quinoline *N*-oxide at the NO group is formed radical $C_9H_7N^{\cdot}$, therewith the oxygen atom eliminates into the structure of $OH^{\cdot}$ radical. The process of desoxygenation is thermally activated, and the formed radical $C_9H_7N^{\cdot}$ readily exerts further fragmentation [2]. Analysis of published data [3] allows to suggest that similar reactions can proceed not only at the protonation of heterocyclic *N*-oxides but also, e.g., at the formation of molecular complexes with hydrogen bond. On the other hand, it is known that in the case of the reactions proceeding with participation of hydrogen atom, in particular of the thermally activated ones, the reaction rate and its direction can be changed at the replacement of a reagent by its deuterated analog (e.g., at the use of D_2O instead of H_2O) [4].

The purpose of this work was the study of the effect of isotopic substitution in water molecule on the physico-chemical properties of quinoline *N*-oxide and estimation of the possibility of proceeding a reaction between the *N*-oxide and deuterated water.

The thermograms of quinoline *N*-oxide crystallohydrates with protic and deuterated water are depicted in Fig. 1.

Let us consider the process of destruction of the crystallohydrate $QO{\cdot}nH_2O$ (Fig. 1). In the first step (20–150°C) the water molecules are removed. The relative mass loss in the first step is about 22.0–22.6% that attests $QO{:}H_2O$ mole ratio equal to 1:2 (Fig. 1a). The water molecules in the crystallohydrate $QO{\cdot}2H_2O$ structure are energetically not equivalent ones as follows from the presence of two peaks on the DTG curve (Fig. 1b), at the temperature 50 and 90°C as well as from the respective peaks on the DTA curve (Fig. 1c). The change in enthalpy at the removing of the first molecule from the crystallohydrate $QO{\cdot}2H_2O \rightarrow QO{\cdot}H_2O + H_2O\uparrow$ is 43.4 ± 0.7 kJ mol^{-1} . On the DTA curve to this process corresponds a relatively strong *endo*-peak (T_{max} = 50°C). The shape of this peak allows to conclude that removing of water from the dihydrate proceeds with melting of the sample. The registered

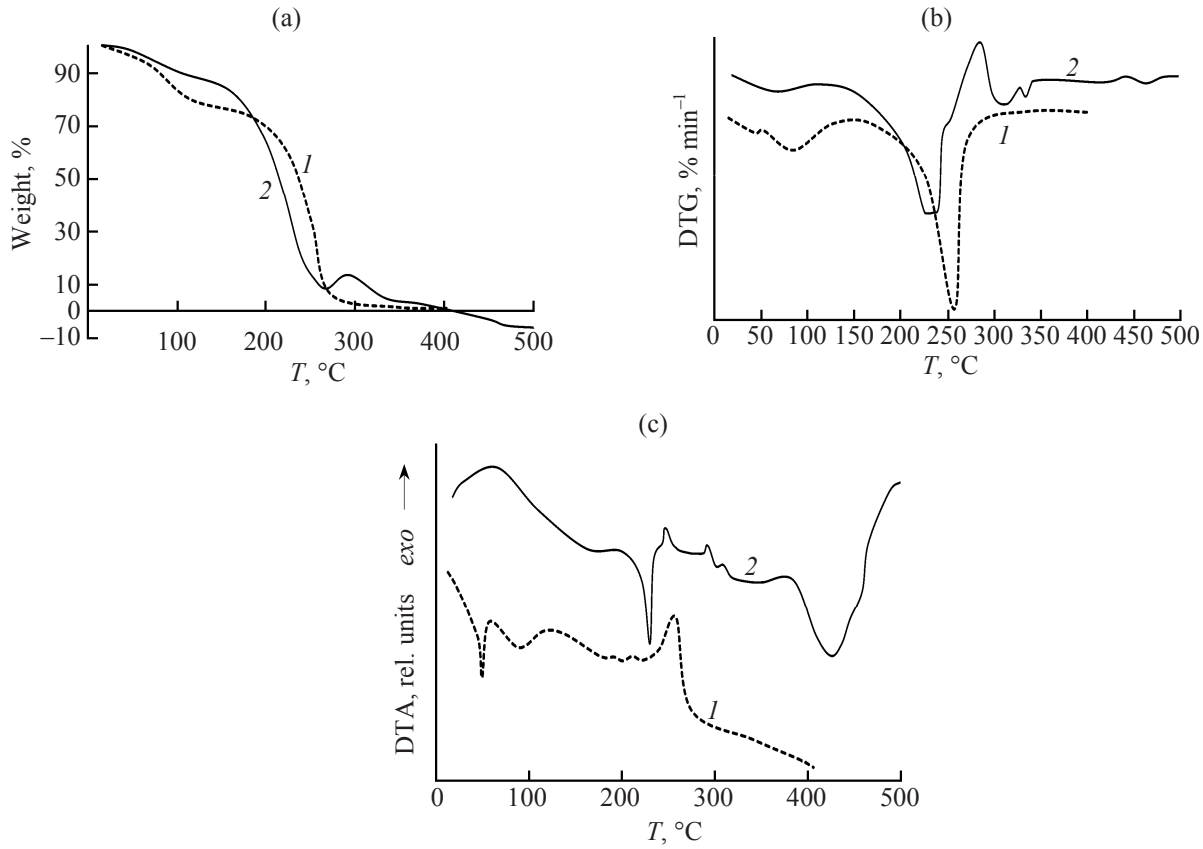
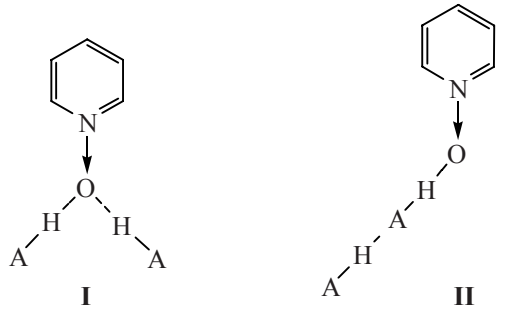


Fig. 1. Thermograms of crystalline samples obtained by quinoline *N*-oxide crystallization from (*1*) H_2O and (*2*) D_2O.

melting point is by ca. 10 K below than the obtained by the authors of [5] that can proceeds from the difference in the conditions of carrying out the experiment. The complete dehydration $QO{\cdot}H_2O \rightarrow QO + H_2O\uparrow$ is characterized by the thermal effect $\Delta H_r(363\ K) = 56.1 \pm 0.8$ kJ mol^{-1}. The obtained values of $\Delta_r H$ that in a first approximation reflect the energy of intermolecular interaction of quinoline *N*-oxide with water molecules attest energetical nonequivalence of the water molecules in the crystallohydrate $QO{\cdot}2H_2O$. It is known that heterocyclic *N*-oxides can interact with protonodonors to form two the most probable structures **I** and **II** [11] (Scheme 1).

Scheme 1.



The structure like **I** has been found in the most cases [12], e.g., in tris[hydrogenbis(pyridine-*N*-oxide)] [13] and in the complex of 2,6-dimethylpyridine *N*-oxide with pentachlorophenol (1:2) [14]. The structure **II** is inherent in the 4-dimethylaminopyridine *N*-oxide dihydrate [15] and 2,2'-bilyridyl-1,1'-dioxide dihydrate [16]. Earlier by the method of X-ray structural analysis of $QO{\cdot}2H_2O$ powder has been established that two water molecules form hydrogen bonds with the oxygen atom of *N*-oxide group [17]. By the data of X-ray structural analysis the lengths of the hydrogen bonds formed between QO and two H_2O molecules are 2.705 Å and 2.601 Å, the oxygen atom of one dihydrate molecule is involved into the hydrogen bonding with the hydrogen atom of another molecule of the dihydrate. Thus, the structural data also clearly indicate energetic nonequivalence of water molecules in the crystallohydrate $QO{\cdot}2H_2O$.

In the Fig. 1a (solid curve) are shown the data obtained for the sample isolated at the crystallization of QO from D_2O. In the first step (20–125°C) occurs dehydration with the mass loss 11.3% of the initial sample weight. The dehydration of the crystalline

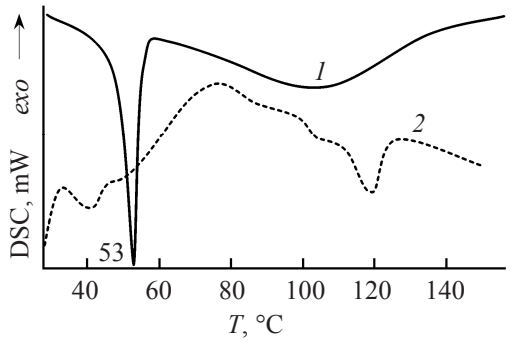


Fig. 2. DSC curves of the samples obtained at quinoline *N*-oxide crystallization from (*1*) H_2O and (*2*) D_2O.

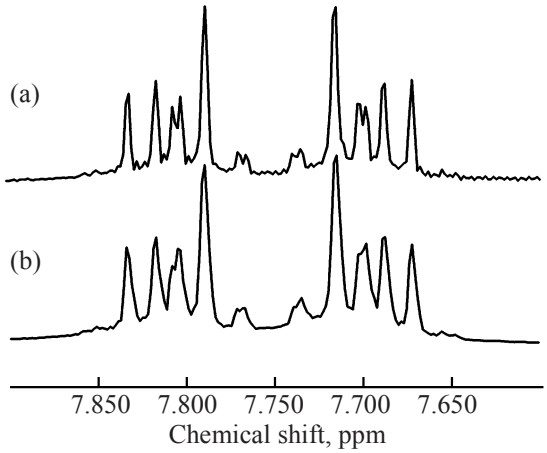


Fig. 3. ^1H NMR spectra. (a) sample obtained by quinoline *N*-oxide crystallization from D_2O (b) phthalimide (Aldrich) in $CDCl_3$, internal reference TMS.

sample obtained at the recrystallization of quinoline *N*-oxide from D_2O proceeds with the thermal effect 70.0 kJ mol^{-1} that is considerably higher that the value of 56.1 kJ mol^{-1} obtained for the $QO \cdot H_2O \rightarrow QO$ process. It should be noted also that within this temperature range on the DTA curve is registered a broad peak responding to an *exo* effect. As far as the crystallohydrate destruction process always proceeds with the energy absorption, hence from the data obtained one can conclude that removing of water either leads to a significant reorganization of the crystal lattice or results in a chemical reaction, therewith the energy gain is over than the energy consumption due to the water removing.

Then at the temperature ~230°C on the DTA curve (Fig. 1c) is registered a narrow peak that corresponds to the process of melting of the sample remaining after D_2O removing. At the attaining temperature 273°C and till to 297°C, on the TG curve is registered increase in the sample mass owing obviously to the oxidation of the reaction product. It should be noted that further the process proceeds with the heat absorption (*endo*-peak on the DTA curve, Fig. 1c) , in distinct to the oxidation of quinoline *N*-oxide obtained from the $QO \cdot 2H_2O$ crystallohydrate (the *exo* peak on the DTA curve, Fig. 1c).

Use of inert atmosphere (N_2) instead of air practically does not affect the process of dehydration, as was judged from the curves of differential scanning calorimetry of the studied samples (Fig. 2).

Thus, the isotopic effect in the studied systems leads not only to the change in the number of the molecules hydrating quinoline *N*-oxide and to the difference in the energetic stability of the crystallohydrates with H_2O and D_2O, but probably also to proceeding a chemical reaction at the heating of quinoline *N*-oxide deuterohydrate.

For the verification of this assumption we carried out spectral analysis of the samples obtained by dissolving quinoline *N*-oxide in the common and in heavy water followed by heating to 100°C, cooling the solution to formation of precipitate, separating the precipitate from the mother liquor and drying it in air. Analysis of ^1H NMR spectra of the samples thus obtained (solvent $CDCl_3$) showed that in the case of D_2O the sample is identified as phthalimide (Fig. 3).

In Fig. 4 are shown typical IR spectra of the samples obtained from QO with H_2O and with D_2O.

The IR spectrum of the crystalline sample obtained with H_2O corresponds to the IR spectrum of quinoline *N*-oxide hydrate, while the sample isolated from D_2O displays spectral characteristics resembling those of isoindolin-1,3-dione (phthalimide) (Fig. 5).

Difference in the spectra in the region above 2000 cm^{-1} (Fig. 5) probably is caused by the isotopic substitution in the phthalimide molecule. To verify this hypothesis we carried out quantum-chemical calculations (Table 1) which showed that the registered spectra correspond to isotope-substituted phthalimide (Scheme 2), and the absorption bands in the region of 2334 and 2442 cm^{-1} correspond to the vibrations of the atomic groups ND and OD, respectively. The observed difference between the measured and calculated values of the

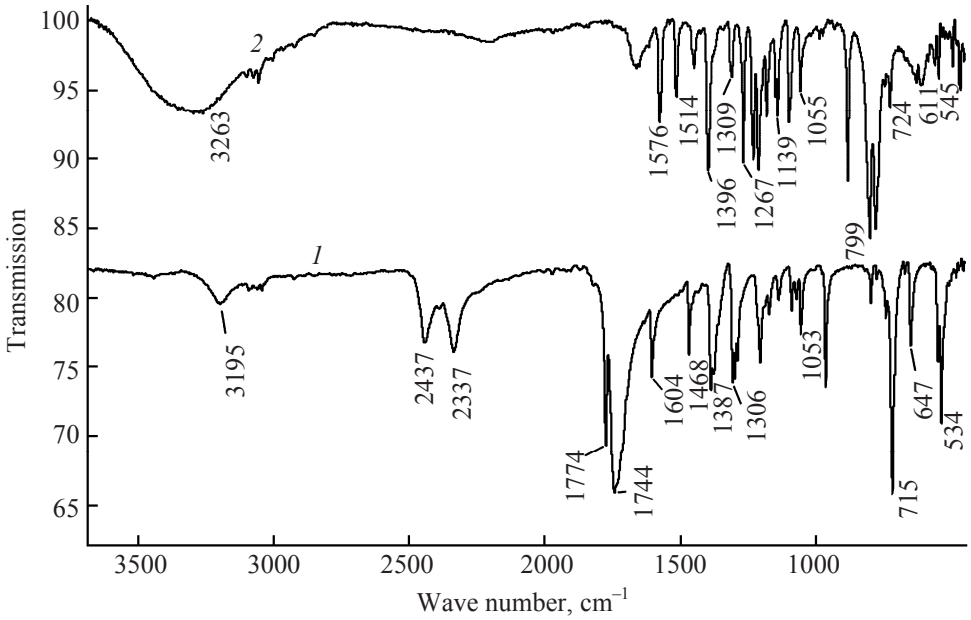


Fig. 4. IR spectra of the samples obtained by quinoline *N*-oxide crystallization from (*1*) H_2O and (*2*) D_2O.

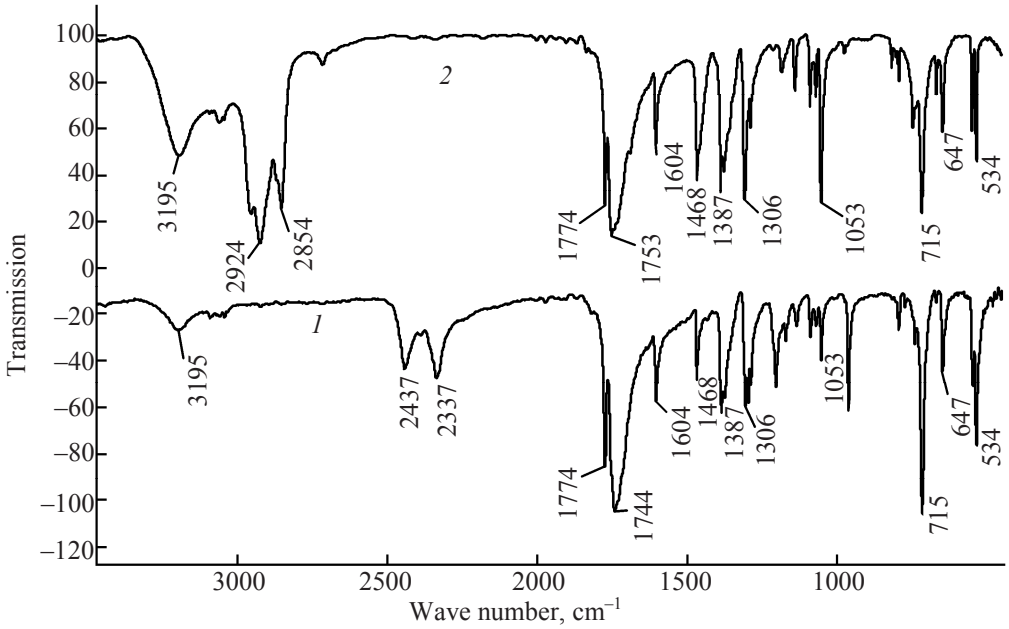
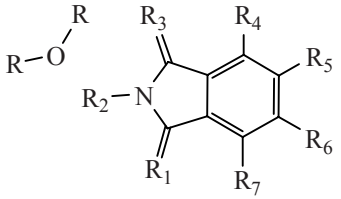


Fig. 5. IR spectra (from KBr pellets) of the sample obtained by quinoline *N*-oxide crystallization from (*1*) D_2O and (*2*) of phthalimide 98% (solid line) [18].

Scheme 2.



Complex $C_8H_2D_3NO_2 \cdot D_2O$: R = R_5 = R_6 = D, R_1 = R_3 = O, R_4 = R_7 = H.

vibration frequency of D_2O molecule caused by the fact that in the real system the D atoms are coordinated by the neighboring molecules. This leads to decrease in the frequency of the bands of symmetric and antisymmetric stretching vibrations of D_2O molecule.

Thus, going back to the thermogram in Fig. 1 we have to talk about phthalimide crystallohydrate with the mole ratio 1:1. It should also be noted that the temperature corresponding to the *endo*-peak at 230°C

Table 1. Experimental and calculated IR spectrum of $C_8H_2D_3NO_2 \cdot D_2O$

Calculated[a]				Experimental
ν, cm^{-1}	intensity, km mol^{-1}	group	vibration mode	ν, cm^{-1}
467.5	199.5	OD	Pendulum	
528.8	12.9	Ph	Deformation	534.2 m
554.1	5	Ph	Deformation	546.7 m
580	35.1	ND	Fan	
616.9	64.3	CD	Fan sym	
640	13.2	Py	Deformation	647.4 m
651.9	10.5	Py	Deformation	669.0 v.w
735	3.2	CD	Pendulum, sym	715.2 v.s
762.4	2.6	ND/Ph	Pendulum, deformation	740.2 w
772.3	24.6	Py	Deformation	772.6 v.w
874.5	1.2	CD	Pendulum, sym	794.1 w
916.1	22	CD	Pendulum, asym	962.0 s
950	2.2	CH	Fan, asym	
952.3	7.5	CH	Fan, asym	1053.0 m
978.1	16.9	Py	Deformation	1069.8 w
1025	1.1	CH	Pendulum, sym	1088.7 m
1153	4.2	Py	Deformation	1134.6 w
1160.6	4.5	Ph	Deformation	1171.3 m
1209.2	44.1	OD	Pendulum sym	1204.1 s
1226.9	87.5	ND	Pendulum	1288.3 s
1315.9	173.5	Py	Stretching	1296.8 s
				1306.1 s
1374.6	80.9	Ph	Stretching	1375.4 s
1417.6	6.8	Ph	Stretching	1386.7 s
1476.2	1	CH	Pendulum sym	1467.7 m
1633	6.7	Ph	Stretching, sym	1604.3 m
1645	16.8	Ph	Stretching, asym	
1774.3	667.4	C=O	Stretching, asym	1742.8 v.s
1835.8	197.7	C=O	Stretching, sym	1773.9 v.s
2529.5	282.6	ND	Stretching	2337.6 br.s
2661.2	125.2	OD	Stretching, sym	2442.3 br.s
2827	59	OD	Stretching, asym	2389.3 m
3193.5	3	CH	Stretching, asym	3092.9 v.w
3194.5	1	CH	Stretching, sym	3200.8 br.w

[a] Vibration with intensity 1.0 or higher. Py is pyrrole fragment, Ph is benzene fragment.

(Fig. 1c) differ from the melting point 238°C found for phthalimide [19], that probably is caused by isotopic substitution in its molecule.

The next step of this work was IR spectral investigation of quinoline *N*-oxide dihydrate, quinoline *N*-oxide monohydrate (the sample heated to 65°C) and quinoline *N*-oxide (the sample after heating to 145–150°C). The results of this part of investigation are listed in Table 2.

The crystalline quinoline *N*-oxide sample heated to 63–65°C is identified spectrally as quinoline *N*-oxide monohydrate (by comparison of its spectrum with the data in the library of low resolution spectra [18]), that is consistent with the data of thermogravimetric alnalysis.

Let us consider the changes in the IR spectra reflecting dehydration of quinoline *N*-oxide.

The absorption band of =C–H vibration in the region of 3100–3040 cm^{-1} depends considerably on the

Table 2. Experimental and calculated IR spectra of quinoline *N*-oxide and related hydrates

Experimental				Calculated			
QO·2 H_2O	QO·H_2O	QO·H_2O [18]	QO	QO			
ν, cm^{-1}				ν, cm^{-1}	intensity, km mol^{-1}	group	vibration mode
563 w	563 w	563 s	559 s	563.1	2.7	Ph	Deformation
582 w	582 w	582 m	583 m	579.6	7.6	Ph	Deformation
626 v.w	628 v.w	627 v.w	626 v.w	598.2	3.3	Ph	Deformation
723 v.w	725 w	725 s	725 s	737.1	4.6	Ph	Deformation
778 v.s	769 v.s	768 v.s	765 v.s	775.2	21.7	CH (Ph)	Fan
799 v.s	797 v.s	796 v.s	796 v.s	802.2	95.7	CH (Py)	Fan
880 v.s	882 s	883 s	882 s	870.4	0.9	CH	Fan
				902.1	11.9	Ph, Py	Deformation
966 v.w	968 v.w	966 v.w	–				
1014 w	1014 w	1014 m	1014 w	1039.5	7.1	Ph	Deformation
1055 m	1057 m	1057 m	1058 m	1078.4	2.4	Py	Deformation
1092 s	1093 s	1097 s	1094 s	1111.5	8.4	CH	Pendulum
1139 s	1137 s	1137 s	1137 s	1160.7	7.0	CH	Pendulum
1180 m	1180 v.w	1180 v.w	–	1167.1	8.2	CH	Pendulum
1210 s	1208 s	1207 m	1210 s	1199.7	14.1	CH	Pendulum
1230 s	1228 s	1229 s	1231 v.s	1234.9	1.9	CH	Pendulum
1266 s	1266 s	1266 s	1265 s	1270.1	19.0	CH, NO	Pendulum, stretching
				1296.7	21.6	CH	Pendulum
1313 m	1308 v.s	1306 v.s	1309 v.s	1350.2	81.9	Ph , NO	Deformation, stretching
1397 v.s	1393 v.s	1393 v.s	1394 v.s	1395.5	2.8	Ph	Deformation
1448 m	1448 m	1447 m	1449 w	1433.5	102.4	CH, NO	Pendulum, stretching
				1473.3	13.8	CH	Pendulum
1514 s	1511 s	1510 s	1512 s	1480.4	6.4	CH	Pendulum
1577 s	1571 s	1570 s	1571 s	1552.8	12.9	Ph, Py	Deformation
				1602.4	37.5	Ph, Py	Deformation
1667 sh.br.m	1655 br.sh.m	1647 ob.br.m	1650 sh. br.m.	1627.3	16.1	Ph, Py	Deformation
				3167.2	0.7	CH (Ph)	Stretching, asym.
3057 w	3068 w	3066 v.w	3007 v.w	3179.6	13.8	CH (Ph)	Stretching, asym.
3076 w			3050 w				
3103 v.w	3101 v.w	3098 v.w	3097 v.w	3184.1	1.5	CH (Py)	Stretching, asym.
				3191.7	17.0	CH (Ph)	Stretching, sym.
				3199.5	9.4	CH (Py)	Stretching, sym.
3293 br.s	3404 br.s	3416 br.s	3418 br.s	3227.6	4.3	CH (Ph)	Stretching, sym.

intermolecular interactions of the heterocyclic *N*-oxide molecules [20]. The band position shift in going from QO (3047 cm^{-1}) and QO×H_2O (3068 cm^{-1}) to QO·2H_2O (3076 cm^{-1}) is defined by increase in the intermolecular π–π- or =C–H···O interactions, that is consistent with the aggregate state of the studied substances: QO and QO·H_2O are liquid while QO·2H_2O is crystalline substance.

The main spectral changes at the hydration of quinoline *N*-oxide are the changes in intensity and position of the bands of stretching vibrations of N–O at ≈1310 cm^{-1}. Intensity of the absorption band at ≈1310 cm^{-1} falls in going from QO to QO·H_2O and further to QO·2H_2O. The hydrogen bonding between the quinoline *N*-oxide N–O group and the proton of water molecule decreases intramolecular charge

transfer and bond order of NO π-bond in the *N*-oxide molecule and finally leads to decrease in intensity of ν(NO) absorption. Note that absorption in the region of 1266 cm^{-1} defined by the vibrations of NO atoms also falls in going from QO to $QO·H_2O$ but on the contrary grows in going from $QO·H_2O$ to $QO·2H_2O$.

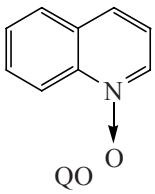
The IR spectra of quinoline *N*-oxide and its hydrates whole resembles each other.

Thus, spectral and thermochenical investigation of quinoline *N*-oxide crystallohydrates with H_2O showed that quinoline *N*-oxide form with H_2O stable dihydrate with energetically nonequivalent water molecules. Complete dehydration of quinoline *N*-oxide occurs at the reaching the temperature 150°C. With accounting for the obtained thermochemical data the individual quinoline *N*-oxide, its monohydrate and dihydrate were isolated and their vibration spectra were studied.

It is established that at the crystallization of quinoline *N*-oxide from D_2O at the heating proceeds a chemical reaction affording isoindoline-1,3-dione. The mechanisms of the reaction of quinoline *N*-oxide transformation into phthalimide at the action of D_2O under mild conditions are not studied yet, but it can be affirmed that the transfer from the sixmembered to the fivemembered ring is unteresting not only in view of fundamental theory but also from the practical viewpoint.

EXPERIMENTAL

Quinoline *N*-oxide (QO) is synthesized and purified according to [5].



QO

The quinoline *N*-oxide crystallohydrates with H_2O and D_2O were prepared by slow crystallization from the respective solvents.

Thermogravimetric analysis of quinoline *N*-oxide crystallohydrates was carried out using thermoanalytical installation described in [6]. The procedure for the treatment of the experimental data of thermogravimetric analysis and determination of physicochemical characteristics of the studied crystallohydrates is described in details in [7–9].

The IR spectrum of the studied crystallohydrates (the ratio sample : KBr = 1:300 wt) was registered on a Avatar 360 FTIR ESP instrument in the frequency range 400–4000 cm^{-1}.

The ^1H NMR spectra of the studied samples and of and phthalimide from Aldrich were registered as solutions in $CDCl_3$ on a pulse spectrometer Bruker AC-200 with operating frequency 200 MHz (^1H). The registration was carried out in the regime of Fourier transformation. A solution was placed to an ampoule of 5 diameter at the temperature 293.15 K. The accuracy of measurements was ±0.0025 ppm.

Differential scanning calorimetry investigations were carried out using a thermochemical unit Setaram TG-DSC 111 under nitrogen atmosphere. Reference compound Pb.

Quantum-chemical calculation of structure and vibration spectra of quinoline N-oxide and phthalimide deuterated monohydrate were carried out by the method of DFT/B3LYP/6-311G** using the program GAUSSIAN 03 [10].

ACKNOWLEDGMENTS

This work was supported by the Presidium of the Russian Academy of Sciences in the framework of the Program of fundamental investigations no. 8 “Development of the methods of synthesis of chemical substances and creation of new materials.”

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