ISSN 1070-3632, Russian Journal of General Chemistry, 2009, Vol. 79, No. 6, pp. 1223–1224. © Pleiades Publishing, Ltd., 2009.
Original Russian Text © R.E. Khoma, A.V. Mazepa, A.A. Shestaka, A.A. Ennann, V.O. Gelmboldt, 2009, published in Zhurnal Obshchei Khimii, 2009, Vol. 79, No. 6, pp. 1048–1049.

## LETTERS TO THE EDITOR

# Condensation of Acetamide in Aqueous Solutions in the Presence of Sulfur(IV) Dioxide

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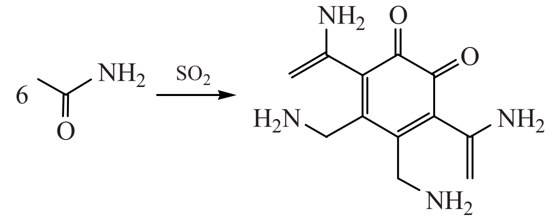
Received December 10, 2008

**DOI:** 10.1134/S107036320906036X

The reactions leading to formation of cyclic structures are not typical for amides, in distinct to their thio analogs that under the conditions of acid catalyzed hydrolysis are prone to cyclization processes with formation of N,S-containing heterocycles [1–3].

We found that reaction of acetamide proceeding in $SO_2–H_3CC(O)NH_2–H_2O$ solution affords cyclic *o*-quinoid compound **I** that formally can be regarded as a product of acetamide condensation along the scheme:



**I**

$+ 4\,H_2O + 2\,NH_3$

In the process of the synthesis of **I** also has been registered formation of acetic acid that can be a result of acetamide hydrolysis in the presence of $SO_2$. Not excluded that the ability of the observed transformation in the presence of $SO_2$ is provided due to weak CH-acid properties of $H_3CC(O)NH_2$ [1].

**4,5-Bis(aminomethyl)-3,6-bis(1-aminovinyl)cyclohexa-3,5-diene-1,2-dione (I).** A mixture of acetamide (0.2 mol) and 3.6 ml of water (0.2 mol) was placed to a reactor, cooled and kept at the temperature 0°C for 20 min. Then through the mixture was passed gaseous $SO_2$ with the rate 10 to 15 ml min$^{-1}$ until homogenous mixture has formed. The viscous oily yellow solution as placed to a freezing chamber (≈ –15°C). After a few weeks the $SO_2$ excess was neutralized at –5 to –10°C by adding $Na_2CO_3$ in small portions until gas evolution stopped. The solution was decanted and again placed into freezing chamber (~ –15°C). After ~14 days the solution obtained was placed to a desiccator over calcium chloride at room temperature and kept until a precipitate has fiormed. We isolated 5.2 g (62 %) of product **I**, white, easily sublimating at room temperature. The IR spectrum, $\nu$, cm$^{-1}$: 3420 m [$\nu_{as}$(NH)]; 3342 s [$\nu_s$(NH)]; 3080 sh [$\nu_{as}(CH_2)(=CH_2)$]; 2930 s [$\nu_s(CH_2)(=CH_2)$]; 2857 s [$\nu_s(CH_2)$]; 1637 m [$\nu$(CO), $\nu$(C=C)]; 1562 s [$\delta(NH_2)$, [$\nu$(C=C)$_{ring}$]; 1453 s [$\delta(CH_2)$, $\delta$(CNH)]; 1397 m [$\delta(CH_2)_{planar.}$]; 1361 m [$\delta(CH_2)$, $r(NH_2)$]; 1071 w [$\nu_{as}$(CN)(C–NH$_{2)}$]; 1061 m [$\delta$(CCC)$_{planar,ring}$ ]; 969 w [$\nu_{ring}$]; 823 m, br [$t(NH_2)$]; 797 m [$\delta(CH_2)$]; 733 m [$\delta$(CCC)$_{out-of-plane,\ ring}$]; 711 m [$r$ (CH$_2$), $t(NH_2)$]; 611 m [$\delta$(CCC)$_{out-of-plane,\ ring}$]; 497 m [$\delta$(OCC)$_{ring}$ ]. The $^1$H NMR spectrum ($CD_3OD$), $\delta$, ppm ($J$, Hz): 1.33–1.37 m (4H, C–N$H_2$), 1.44–1.53 m (4H, CH$_2$–N$H_2$) 2.63 t (4H, C$H_2$–NH$_2$, $J_{HH}$ 7.2), 4.87 s (4H, C=C$H_2$). The mass spectrum (FAB, solution of I in 3-nitrobenzyl alcohol), $m/z$ ($I_{rel}$ %): 250 (100) $[M + 2H]^+$; (FAB, solution of **I** in 3-nitrobenzyl alcohol with NaI adding) $m/z$ ($I_{rel}$, %): 271 (65) $[M + Na]^+$, 250 (53) $[M + 2H]^+$. In the FAB mass-spectrum occurs a peak of $[M + 2H]^+$, characteristic of *o*-quinones that are reduced to the corresponding catechols, that is not inherent in *p*-quinones [5]. Found, %: C 57.43; H 6.41;

N 23.21. $C_{12}H_{16}N_4O_2$. Calculated, %: C 58.05; H 6.50; N 22.57. *M* 248.3.

The carbon and hydrogen contents were analyzed on a CHN-analyzer. The IR spectra were recorded on a Spectrum BX II FT-IR System instrument (Perkin-Elmer) in the range 4000–350 cm$^{-1}$, the samples were prepared as pellets with KBr. The $^1$H, $^{13}$C NMR spectra were registered on a Bruker WM 400 spectrometer. The FAB mass spectra were registered on a VG 7070 instrument, desorption of ions from a liquid matrix was carried out by the action of the beam of argon atoms with the energy 8 keV.

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