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SHORT COMMUNICATIONS

Revised Structure Determination of the Schiff Base Derived from Tricyclo[7.3.1.0^{2,7}]tridec-2(7)-en-13-amine and Salicylaldehyde

O. V. Patrusheva and V. I. Vysotskii

Far-East State University, ul. Oktyabr'skaya 27, Vladivostok, 690600 Russia
e-mail: vlad@chem.dvgu.ru

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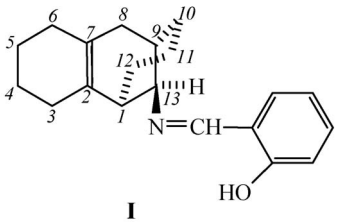
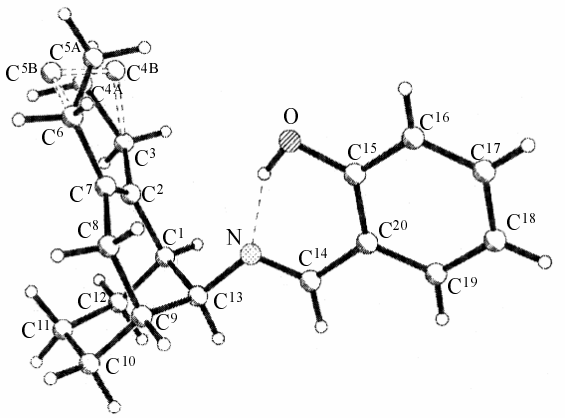
In the previous communication [1] we described the structure of Schiff base **I**, which was synthesized from the *syn* isomer of tricyclo[7.3.1.0^{2,7}]tridec-2(7)-en-13-amine and salicylaldehyde. The results of X-ray diffraction study of compound **I** at 295 K showed high thermal parameters of the C^4 and C^5 atoms, and the C^4–C^5 distance was estimated at 1.348 E, which corresponds to a double C=C bond length. However, neither the synthetic procedure nor the data of other physical methods gave grounds to assume the presence of a double bond in the above position. In order to elucidate reasons for such inconsistency, we have determined the structure of Schiff base **I** at 173 K [2] (see figure).



I

We have found molecule **I** in crystal is statistically disordered due to the presence of two conformers, which leads to a reduced (averaged) length of the C^4–C^5 bond. This pattern is observed for the first time in the series of tricyco[7.3.1.0^{2,7}]tridecane derivatives. The structure was solved by the direct method and was refined by the least-squares procedure in anisotropic approximation for non-hydrogen atoms. The positions of hydrogen atoms were determined by the difference synthesis of electron density and were refined in isotropic approximation. The C^4 and C^5 atoms are statistically disordered by two positions, , respectively. The populations of these positions are 0.9 (*A*) and 0.1 (*B*). The hydrogen atoms on C^{4B} and C^{5B} were not localized by the difference synthesis and were not included into the subsequent refinement.



Structure of the molecule of *N*-salicylidenetricyclo-[7.3.1.0^{2,7}]tridec-2(7)-en-13-amine (**I**) at 173 K.

The crystalographic data were acquired and the unit cell parameters were refined using SMART and SAINT Plus software [3]. All calculations were performed with the aid of SHELXTL/PC software [4].

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REFERENCES

1. Vysotskii, V.I., Patrusheva, O.V., Vysotskaya, T.A., and Isakov, V.V., *Russ. J. Org. Chem*., 2002, vol. 38, p. 1130.
2. Cambridge Crystallographic Data Center, Deposition no. 206 318.
3. *SMART and SAINT-Plus. Versions 5.0. Data Collection and Processing Software for the SMART System*, Madison, Wisconsin, US: Bruker AXS, 1998.
4. *SHELXTL/PC. Versions 5.10. An Integrated System for Solving, Refining, and Displaying Crystal Structures from Diffraction Data*, Madison, Wisconsin, US: Bruker AXS, 1998.

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