

VIBRATION SPECTRA OF 2-[(TRICHLOROACETYL) AMINO]BENZAMIDE AND A PARTIAL ASSIGNMENT OF THE VIBRATIONAL BANDS

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ABSTRACT

The FTIR Absorbance, FTIR ATR, DRIFT and Raman spectra of 2-[(trichloroacetyl)amino]benzamide have been registered in solid state at resolution of 1 cm⁻¹. Quantum chemistry calculations were performed by using the Gaussian 98, Revision A.7. For the geometry optimization and vibrational spectra prediction the B3LYP density functional with 6-31G(d) basis set was used. A partial assignments of vibrational bands is given. FTIR Absorbance, FTIR ATR, DRIFT and Raman spectra of 2-[(trichloroacetyl)amino]benzamide **1** are given as a Supplementary Material in JCAMP-DX format, version 4.24.

Keywords: FTIR; Raman Spectroscopy; 2-[(trichloroacetyl)amino]benzamide

INTRODUCTION

Number of representatives of 2,4(1*H*,3*H*)-quinazolinediones have shown a wide spectrum of biological activities. Developed by us method for the synthesis of 3-substituted 2,4(1*H*,3*H*)-quinazolinediones includes intramolecular cyclization of 2-[(trichloroacetyl)amino]benzamides [1]. In the course of elucidation of this interaction, a necessity of study arises concerning the influence of N-(un)substituted benzamido group on the proceeding of the reaction. For this purpose an appropriate for investigation chemical structure is 2-[(trichloroacetyl)amino]benzamide.

EXPERIMENTAL

The FTIR Absorbance, FTIR ATR, DRIFT and Raman spectra of 2-[(trichloroacetyl)amino]benzamide **1** have been registered in solid phase at resolution of 1 cm⁻¹. The three types of IR spectra were recorded on a VERTEX 70 (Bruker, Germany). The used ATR accessory is MIRacle™ (ZnSe crystal, produced by PIKE Technologies, Inc), and the Diffuse Reflection Accessory is Praying Mantis™ (produced by Harrick Scientific Products, Inc.). The Raman spectrum is measured on RAM II (Bruker, Germany) with a focused laser beam of 100 mW power of Nd: YAG laser (1064 nm).

Quantum chemistry calculations were performed by using the Gaussian 98, Revision A.7 [2]. For the geometry optimization and vibrational spectra prediction the B3LYP density functional with 6-31G(d) basis set was used.

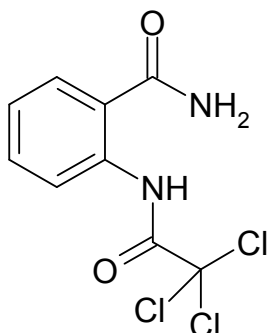


Figure 1. *Chemical structure of 1.*

RESULTS AND DISCUSSION

The registered FTIR and Raman spectra are given in Fig. 2.

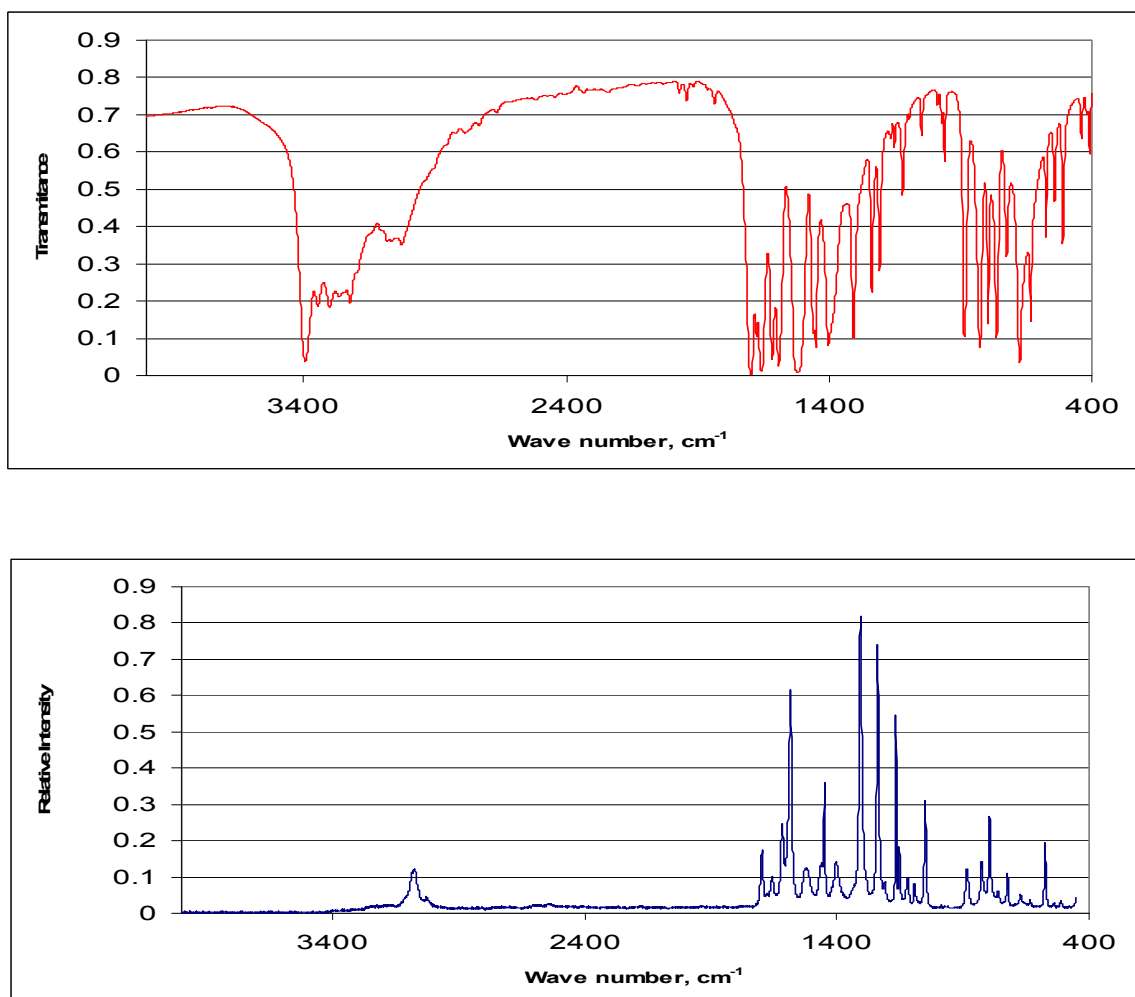


Figure 2. *The infrared and Raman spectra of 1.*

The comparison of vibrational spectra of **1** with those of N-(4-chlorobenzyl)-2-[(trichloroacetyl)amino]benzamide and the performed quantum chemistry calculations permit an assignment for some of the spectral bands of **1**. The proposed assignments are the following: valence $\nu_{\text{as}}(\text{NH}_2) = 3395 \text{ cm}^{-1}$, $\nu_{\text{s}}(\text{NH}_2) = 3302 \text{ cm}^{-1}$, $\nu(\text{NH}) = 3224 \text{ cm}^{-1}$. Stretching C-H vibrations in benzene ring are 3106 cm^{-1} , 3084 cm^{-1} , 3067 cm^{-1} and 3028 cm^{-1} . Stretching carbonyl vibrations are 1698 cm^{-1} (trichloroacetamido group) and 1657 cm^{-1} . Benzene ring vibrations are: 1584 cm^{-1} (8a) 1307 cm^{-1} (14) and 1048 cm^{-1} (18a), and C-H out-of-plane bending of the benzene ring is at 763 cm^{-1} (ortho substituted benzene).

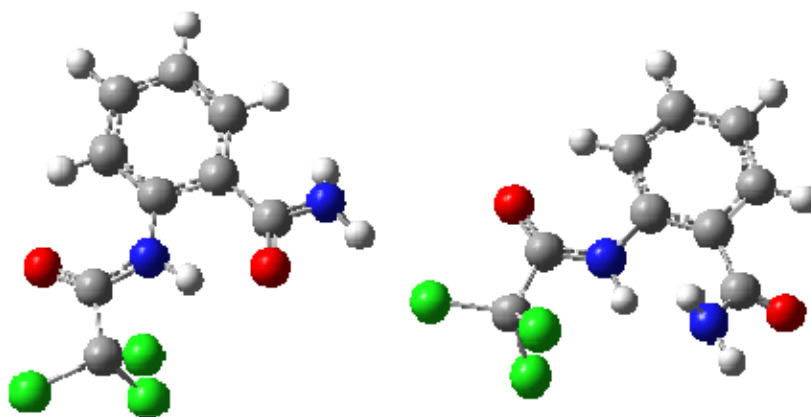


Figure 3. *The conformers of 1 optimized by quantum chemistry calculations.*

There appear 5 bands and a shoulder in the N-H stretching region of the IR spectrum. The quantum chemical calculations show the existence of two stable conformers – one with a hydrogen bond between N-H and C=O, the other with a hydrogen bond between N-H and the nitrogen of the NH_2 , see Fig.3. The three bands given above correspond to the second structure. The position of the others is 3347 cm^{-1} , 3266 cm^{-1} and 3195 cm^{-1} (shoulder). The weak band at 1677 cm^{-1} supports the presence of a second conformer: it can be assigned to the C=O stretching mode of benzamido group.

SUPPLEMENTARY MATERIAL

FTIR Absorbance, FTIR ATR, DRIFT and Raman spectra of 2-[(trichloroacetyl)amino]benzamide **1** are given as a Supplementary Material in JCAMP-DX format, version 4.24 at <http://www.kosnos.com/spectroscopy/iris>.

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REFERENCES

1. Petrov J. S., Andreev G. N.; Synthesis of 2,4(1*H*,3*H*)-quinazolinedione and 3-substituted 2,4(1*H*,3*H*)-quinazolinediones. *Org. Prep. Proced. Int.*, 37 (2005) 560-565.
2. Gaussian 98, Revision A.7, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, V. G. Zakrzewski, J. A. Montgomery, Jr., R. E. Stratmann, J. C. Burant, S. Dapprich, J. M. Millam, A. D. Daniels, K. N. Kudin, M. C. Strain, O. Farkas, J. Tomasi, V. Barone, M. Cossi, R. Cammi, B. Mennucci, C. Pomelli, C. Adamo, S. Clifford, J. Ochterski, G. A. Petersson, P. Y. Ayala, Q. Cui, K. Morokuma, D. K. Mallick, A. D. Rabuck, K. Raghavachari, J. B. Foresman, J. Cioslowski, J. V. Ortiz, A. G. Baboul, B. B. Stefanov, G. Liu, A. Liashenko, P. Piskorz, I. Komaromi, R. Gomperts, R. L. Martin, D. J. Fox, T. Keith, M. A. Al-Laham, C. Y. Peng, A. Nanayakkara, C. Gonzalez, M. Challacombe, P. M. W. Gill, B. Johnson, W. Chen, M. W. Wong, J. L. Andres, C. Gonzalez, M. Head-Gordon, E. S. Replogle, and J. A. Pople, Gaussian, Inc., Pittsburgh PA, 1998.