研究简报

3-氨基-酪氨酸盐酸盐的合成与晶体结构

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The Synthesis and Novel Structure of the Hydrochloride of 3-aminotyrosine

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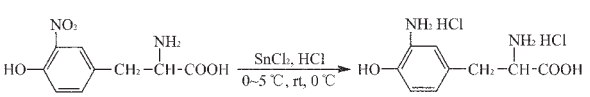
Colorless single crystal of the title compound was accidentally obtained by a reaction of 3-nitrotyrosine with chloride stannous at different temperature over more than two months. In the structure of hydrochloride of 3-aminotyrosine, the strong interaction of hydrogen-bonds results in the formation of 3D network. CCDC: 251940.

Keywords: **tyrosine** **crystal structure** **hydrogen-bond**

Comment

The biological relevance of tyrosine nitration is a subject of much interest, because extensive evidence supports formation of 3-nitrotyrosine in vivo under a variety of different pathological conditions. Nitrated tyrosine residues are widely considered as biomarkers for the involvement of reactive species derived from nitrogen monoxide in a variety of pathophysiological conditions such as neurodegenerative diseases, atherosclerosis, bacterial and viral infections, chronic inflammation, and cancer[1]. Our work focuses on the capture of the reduction intermediate of nitrotyrosine when used different reductant (such as different Lewis acid). But to our surprising, we got different results[2-4]. And in this paper we report on the synthesis and crystal structure of hydrochloride of 3-aminotyrosine (**1**), which, to the best of our knowledge, has not been reported before. The title compound was accidentally obtained by a reaction of 3-nitrotyrosine with chloride stannous at different temperature over more than two months (scheme 1).



Scheme 1

In the crystal structure of the title compound, there is one lattice water molecule, two chloride anions and the cation of 3-aminotyrosine with two charge. The two chloride anions, the O-H of one water molecule, the H atoms of the hydroxyl group and the O-H of the carboxyl group as well as the H atoms of the amino group all involve in hydrogen-bonds forma-

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tion. And the strong interaction of hydrogen-bonds results in the formation of 3D network. The distance of N-C in this compound is a little different, the distance of N(2)-C(8) is 0.146 1(3) nm shorter than that found in literature, while the distance of N(1)-C(3) is 0.149 9(3) nm which is much longer than the average (0.148 3 nm)[5]. The distance of O(1)-C(2) is 0.132 4(3) nm longer than the distance of O(2)-C(2) which is 0.120 0 (3) nm, this can also indicates that the carboxyl group in the title compound is neutral.

Experimental

25 g *L*-3-nitrotyrosine[6] was added to the hydrochloric acid solution of 7.5 g $SnCl_2 \cdot 2H_2O$ at 0 ℃. The mixture stirred at room temperature until it turned clear. More than two months later, colorless single crystals were formed from the freeze clear solution in the refrigerator. Intensity data were collected at 293(2) K on a Bruker AXS SMART CCD for a dimension of 0.10 × 0.15 × 0.18 mm^3. $C_9H_{16}Cl_2N_2O_4$, M=287.14, space group $P2_1$ with a=0.508 75(10) nm, b=1.679 6(3) nm, c=0.789 93(16) nm, $\alpha=\gamma=90°$, β=91.40(3)°, V=0.674 8(2) nm^3, Z=2, R_1 [$I>2\sigma(I)$]=0.032 1, wR_2(all)=0.102 4. Flack=0.05(5). Programs used: SAINT, SADABS, SHELX-97, and ORTEP.

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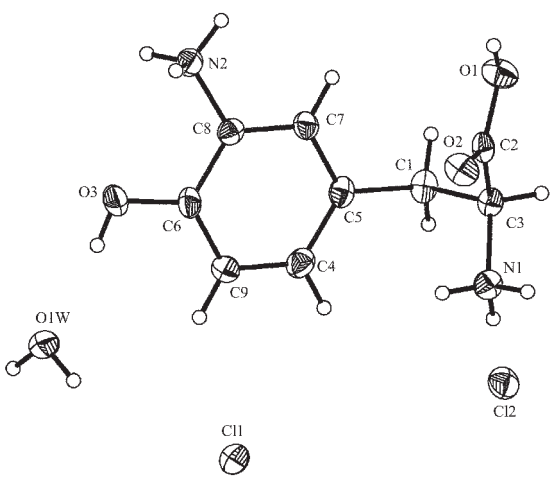

Fig.1 ORTEP diagram (50% probability ellipsoids) showing the solid-state structure for **1**

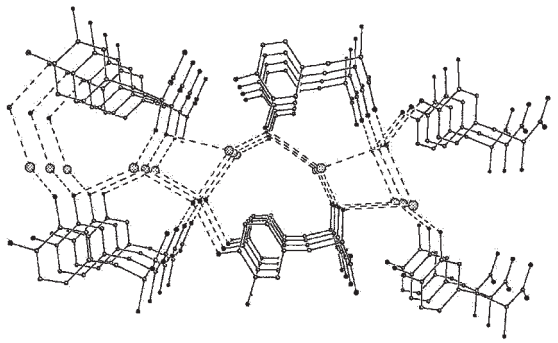

Fig.2 A perspective view of the 3D network of **1** through H-bonds

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[6] *L*-Tyrosine 14.5 g in 60 mL H_2O treated during 0.5 h at room temperature with 15 mL 50% HNO_3 cooled to 5~8 ℃ treated during 2 h with an addition 32 mL 50% HNO_3 stirred 5 h at room temperature, refrigerated overnight and filtered, yield 5.1 g. *L*-3-nitrotyrosine.