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[Ni(NH_2NHCO_2CN_3)_3](NO_3)_2·H_2O的制备、具体结构和热分解机理

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摘要 由硝酸镍水溶液和肼基甲酸甲酯（NH_2NHCOOCH_3, MCZ）的水溶液反应，制备出未见文献报道的配合物[Ni(MCZ)_3]-(NO_3)_2·H_2O。晶体结构测定结果表明， 该晶体属单斜晶系，P2_1/n空间群，晶体学参数为：a = 1.3681 (2) nm，b = 0. 8188 (1) nm，c = 1.6029 (4) nm，β = 92.16 (2)°，V = 1.7943 (6) nm~3， D_c = 1.744 g·cm~(-3)，Z = 4，F(000) = 976，μ(Mo Kα) = 1.166 mm~(-1) 。结构采用全矩阵最小二乘法优化，除氢原子采用各向同性热参数外，其它非氢原子均采用各向异性热参数修正，最终偏离因子R_1 = 0.0337，wR_2 = 0.0857。在 该配合物分子中，肼基甲酸甲酯作为双齿配体，由羰基氧原子和端基氮原子与Ni~ (2+)配位，形成五元平面螯合环，配合物分子中共有三个这样的螯合环，中心离子为六配位八面体构型。配合物的外界是两个硝酸根离子和一个水分子，通过库仑力和氢键与内界结合在一起。采用TG-DTG，DSC，IR等表征了标量化合物的热稳定性 。在程序升温条件下，该配合物的热分解过程是由一个弱的吸气热过程和三个较强 的连续的放热过程组成的，由TG-DTG和IR分析结果证明，在325 ℃时的最终分解产 物为NiO，得到了化合物的分解机理。

关键词 肼 甲酸酯 硝酸镍 晶体结构 热解 示差扫描量热法 红外分光光度法

分类号 0621

Preparation, Crystal Structure and Thermal Decomposition Mechanisms of [Ni (NH_2NHCO_2CH_3)_3] (NO_3)_2·H_2O

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Abstract The title compound [Ni(MCZ)3J (NG^^" H2O was prepared by reaction of the aqueous solutions of nickel nitrate and methylcaxbazate (MCZ) . The crystal structure of [NKMCZ^KNO.^'EkO was determined by using single crystal diffraction techniques and crystalline was in monoclinic system with space group P2\/n. The unit cell parameters are as follows: o = 1.3681(2) nm, 6=0.8188(1) nm, c = 1.6029(4) nm, β= 92. 16(2)°, V= 1.7943(6) nm~3, Z = 4, F(000) =976, D_c= 1.744 g·cm~(-3), μ(Mo Kα) = 1.166 mm"1. Positional and thermal parameters were refined by the full-matrix least-squares method. All the non-hydrogen atoms were refined anisotropically but the hydrogen atoms were refined isotropically with the final factor values of R_1 = 0.0337 and wR_2 = 0.0857. In the title compound, methylcaxbazate serves as a bidentate ligand coordinating to the nickel cation with oxygen atom from carbonyl and the nitrogen atom from hydrazine group so that a five- membered chelating ring is formed. There are three rings in the molecular structure. The coordination number is six and the configuration is octahedral. In the environment of the molecule there are two NO_3~- and one crystal water molecule, which are linked with the inside by electrostatic force and hydrogen bonds. TheiTnal decomposition mechanisms of the compound were characterized by using TG-DTG, DSC and IR techniques. It was confirmed that decomposing processes of the compound include one endothermal and three continuous exothermic stages, residue is NiO at 325 ℃ , finally.

Key words HYDRAZINE FORMIC ESTER NICKEL NITRATE CRYSTAL STRUCTURE THERMOLYSIS DSC IR

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