New bisphosphinamide: X-ray crystallography, spectroscopy and thermogravimetry

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Phosphinamides, with a $C_2P(O)N$ fragment, are well-known for flame-retardancy properties [1]. In this work, the synthesis, spectroscopy, thermogravimetry and X-ray crystallography of N, N'-(cyclohexane-1,4-diyl)-bis(P, P-diphenylphosphinic amide) are studied. The compound crystallizes in the monoclinic space group *C*2/*c*. The asymmetric unit is composed of one half of the molecule, and the complete molecule is generated by an inversion element. The P atom has a distorted tetrahedral $C_2P(O)N$ enviroment, and the maximum and minimum bond angles at phosphorus are related to N—P═O (120.23 (6)°) and N—P—C (101.95 (7)°). The bond-angle sum at nitrogen (about 353°) confirms its sp^2 character. The P═O bond length (1.4864 (11) Å) is slightly longer than the typical phosphorus-oxygen double bond length and is comparable to those in analogous compounds [2, 3]. The P—N bond length (1.6407 (13)Å) is standard for structures with a $C_2P(O)N$ skeleton [3]. The NH unit adopts a *synclinal* orientation with respect to the P═O group. In the crystal structure, the molecules are aggregated through N—H…O hydrogen bond in a two dimensional assembly along the *bc* plane. The ^{31}P NMR spectrum shows one signal at 19.91 ppm indicating the high purity of the compound. In the IR spectrum, the band centered at 3133 cm^{-1} corresponds to the N—H stretching. The TGA exhibits the starting decomposition point at about 250 °C.

Keywords: Bisphosphinamide, Crystal Structure, Thermogravimetric Analysis

References

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