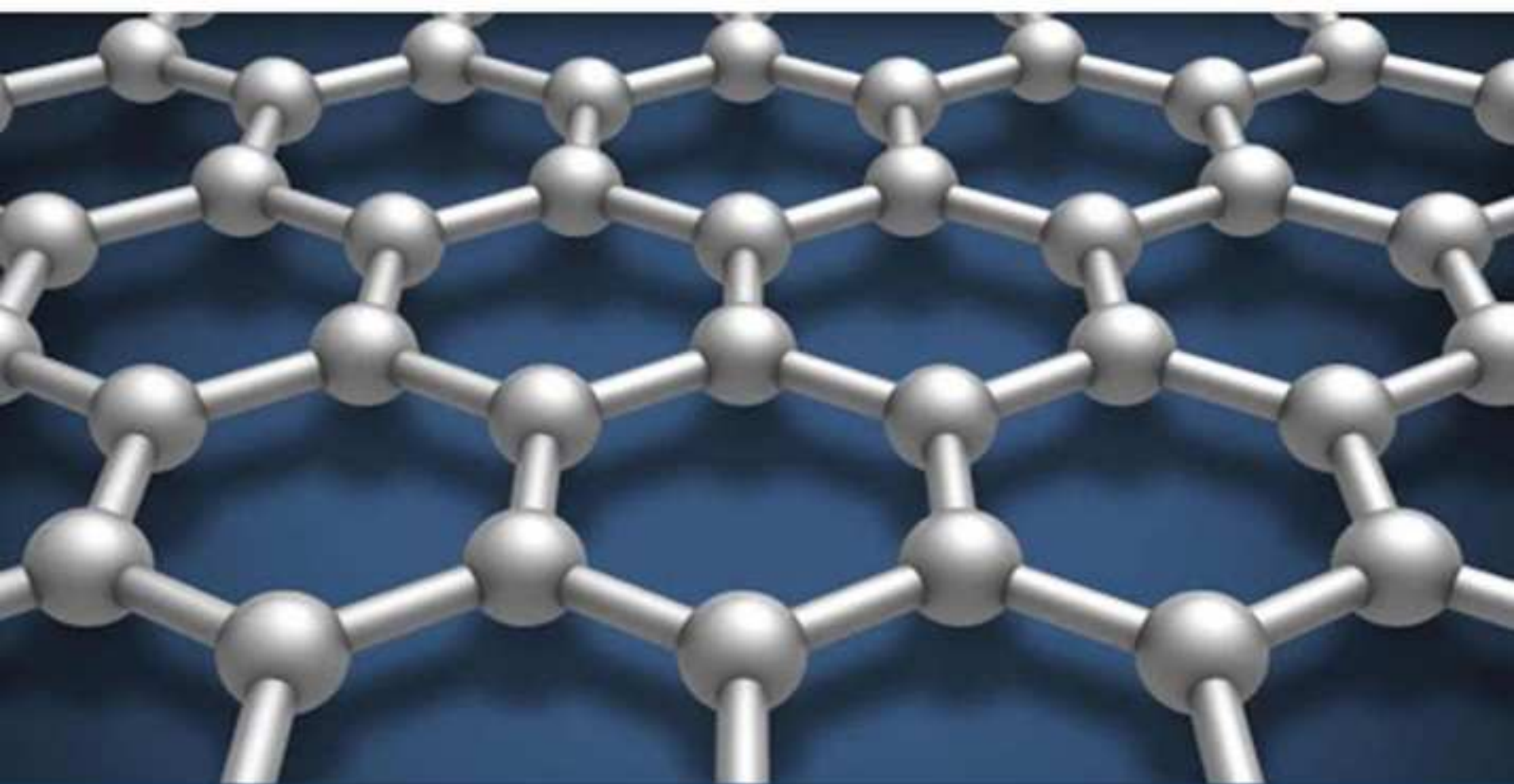


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Узбекский научно-технический и производственный журнал

Композиционные материалы

деформации. Приняты гипотезы об абсолютной гибкости нитей и достаточной малости коэффициентов внутреннего трения. Получена

система уравнений статики, в составе которой: уравнения равновесия, уравнения, связывающие перемещения и деформации каждого

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NONLINEAR OPTICAL (NLO) MATERIALS: BIS(2-METHYL-4-NITROANILINIUM) HEXACHLORO-TIN(IV) MONOHYDRATE. QUANTUM CHEMICAL CALCULATIONS. SYNTHESIS, CRYSTAL STRUCTURE, HIRSHFELD SURFACE ANALYSIS, CRYSTAL VOIDS.

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Abstract. 2-Methyl-4-nitroaniline (2m4na) and Tin(IV) chloride formed a nonlinear optical material. The molecule obtained nonlinear optical material(NLO)=(2m4na)₂SnCl₆·H₂O-bis(2-Methyl-4-nitroanilinium)hexachloro-Tin(IV) monohydrate. In all the crystal structures presented, cations and anions are arranged alternatively to form chain and ring hydrogen-bonding patterns consisting of weak unconventional N–H...Cl hydrogen bonds. The compound was first analyzed using single-crystal X-ray diffraction to determine its crystal structure. Subsequently, FTIR (Fourier-transform infrared spectroscopy) and UV-visible absorption spectroscopy studies were conducted to provide insights into the compound's chemical composition and electronic transitions. Nonlinear optical materials were studied by Hirshfeld surface analysis and crystal void parts.

Key words: 2-Methyl-4-nitroaniline; Single crystal; Hydrogen bond; Hirshfeld surface analysis; Crystal Voids.

Introduction. The design and synthesis of new organic-inorganic hybrid compounds have become significantly important in recent years due to the potential to merge the distinct properties of organic and inorganic materials[1]. The self-assembly processes of these hybrid compounds in the solid-state are facilitated by a diverse array of interactions, including hydrogen bonding networks, π - π interactions, and van der Waals forces. These interactions play a crucial role in governing the supramolecular organization and properties of the resulting materials[2]. The hydrogen bonding network contributes to the structural stability and organization of the hybrid compounds. π - π

interactions, which involve the stacking of aromatic systems, can influence the electronic properties and structural arrangement of the materials[3;4]. Additionally, van der Waals forces contribute to the overall cohesion and packing efficiency of the hybrid structures [5].

Results and discussion. 1. Single crystal X-ray structure analysis. The intermolecular hydrogen bonds and hydrogen bonds in the molecule are between the N1-H atom in 2-methyl 4-nitro aniline and the Cl2 and Cl3 atoms in SnCl₄. The intermolecular bond is N1-H...Cl3 and N1-H...Cl2 occurs between Cl2 and between N1-H...H-O hydrogen bonds. The intermolecular bond

is N1-H...Cl2 distance is 2.579 Å and distance of N1-H...Cl2 is 2.883 Å. The valence angle of Cl-Sn-Cl in the SnCl_6^{2+} molecule is 92.50 Å.

Information of plane spacing for monoclinic structure crystal systems.

$$\frac{1}{d^2} = \frac{1}{\sin^2 \beta} \cdot \left(\frac{h^2}{a^2} + \frac{k^2 \sin^2 \beta}{b^2} + \frac{l^2}{c^2} - \frac{2hl \cos \beta}{ac} \right)$$

Additionally, corrections for Lorentz and polarization effects were made to ensure the accuracy of the intensity measurements of the reflections. Monoclinic, Axial lengths and angles - Three unequal axes, one pair not at right angles $a \neq b \neq c$, $\alpha \neq \gamma = 90 \neq \beta$ [6;7].

The X-ray diffraction data for the crystal structure of 2-Methyl-4-nitrobenzenaminium hexachloro-stannane monohydrate was collected using a KUMA Diffraction KM-4 four-circle single-crystal diffractometer [8]. (Fig-1).

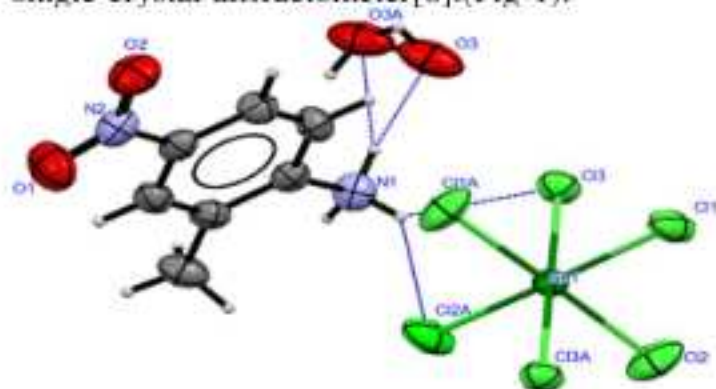


Fig-1. (a) ORTEP drawing of the intermolecular H-bonds and (b) Nonlinear optical (NLO) materials.

bis(2-Methyl-4-nitroanilinium) hexachloro-Tin(IV) monohydrate molecule. Thermal ellipsoids are drawn with the 50% probability level.

This instrument was equipped with a CCD detector and utilized graphite-monochromatized Mo K α radiation with a wavelength of 0.71073 Å.

The raw data obtained from the diffraction experiment were processed using the CrysAlis Data Reduction Program, specifically version. During the data reduction process, an absorption correction was applied to account for any absorption effects present in the crystal sample [9;10].

The crystal structures of 2-Methyl-4-nitrobenzenaminium hexachloro-stannane monohydrate were initially solved using direct methods. Subsequently, the structures were refined utilizing the full-matrix least-squares method with the SHELXL-97 software [11]. This refinement process allowed for a detailed and precise analysis of the crystal structure, ensuring that the final model accurately represented the arrangement of atoms within the compound [12].

The crystallographic data for 2-methyl-4-nitroanilinium chloride (1) can be accessed through CCDC-875783 and for bis(2-amino-4-nitroanilinium) hexachloridostannate(IV) monohydrate (2) through CCDC-905995. Supplementary data related to the crystallography in this study can be acquired at no cost from www.ccdc.cam.ac.uk/conts/retrieving.html or by contacting the Cambridge Crystallographic Data Centre at 12 Union Road, Cambridge CB21EZ, UK; fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk.

Crystallographic data and details of the structure refinement are given in Table 1.

Table 1

Crystallographic data and details of the refinement of the bis(2-Methyl-4-nitroanilinium) hexachloro-tin(IV) monohydrate. structure.

Name	bis(2-Methyl-4-nitroanilinium) hexachloro-tin(IV) monohydrate.
Empirical formula	$2(\text{C}_7\text{H}_9\text{N}_2\text{O}_2^+)\text{Cl}_6\text{Sn}^{2+} \cdot \text{H}_2\text{O}$
Formula weight	655.71
Temperature/K	295(2)
Crystal system	Monoclinic
Space group	C2/c
a/Å	12.5392(3)
b/Å	9.2956(2)
c/Å	20.6018(4)
$\alpha/^\circ$	90
$\beta/^\circ$	99.435(2)
$\gamma/^\circ$	90
V/Å ³	2368.85
Z	1
ρ_{calc} , g/cm ³	1.342
μ/mm^{-1}	0.895
F(000)	848.0
Crystal size/mm ³	0.16 × 0.14 × 0.12
Radiation	Cu K α ($\lambda = 1.54184$)
2 θ range for data collection/ $^\circ$	8.13 to 152.25

Index ranges	$100 \leq h \leq -100, 100 \leq k \leq -100, 100 \leq l \leq -100$
Reflections collected	13000
Independent reflections	2045 [$R_{\text{int}} = 0.0297, R_{\text{sigma}} = 0.0159$]
Data/restraints/parameters	2045/0/180
Goodness-of-fit on F^2	1.055
Final R indexes [$I > 2\sigma(I)$]	$R_1 = 0.0343, wR_2 = 0.0940$
Final R indexes [all data]	$R_1 = 0.0372, wR_2 = 0.0969$
Largest diff. peak/hole / $e \text{ \AA}^{-3}$	0.20/-0.15
R-Factor(%)	2.06
Reduced Cell Parameters	a: 7.804 b: 7.804 c: 20.602

The transitioning to the compound bis(2-Methyl-4-nitroanilinium) hexachloro-Tin(IV) monohydrate, this complex compound consists of the cation bis(2-Methyl-4-nitroanilinium) and the anion hexachloro-Tin(IV) along with a water molecule forming a monohydrate. In the crystal structure of bis(2-Methyl-4-nitroanilinium) hexachloro-Tin(IV) monohydrate, the packing of molecules will be influenced by the interactions between the cation, anion, and the water molecule. The coordination number of Tin(IV) in this

compound is typically 6, indicating that Tin(IV) is surrounded by six chloride ions (Fig-2).

In the crystal structure of 2-Methyl-4-Nitroanilinium Hexachloridostannate(IV), the layers are arranged in an ACC sequence due to the bivalent SnCl_6^{2-} anion balancing two monovalent $2\text{m}4\text{na}^+$ ions.

The aromatic rings of adjacent $2\text{m}4\text{na}^+$ ions are parallel to each other, as well as to the ions in the next layer. However, the vectors of neighboring ions are close to orthogonal, creating a unique geometric relationship.

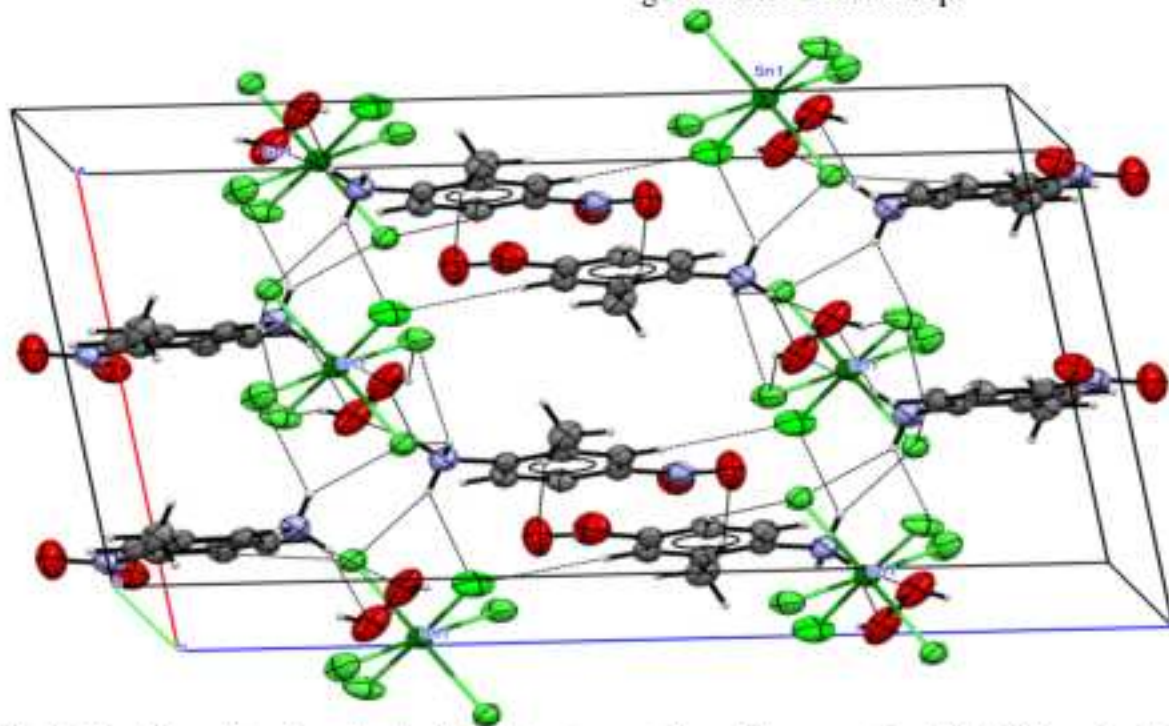


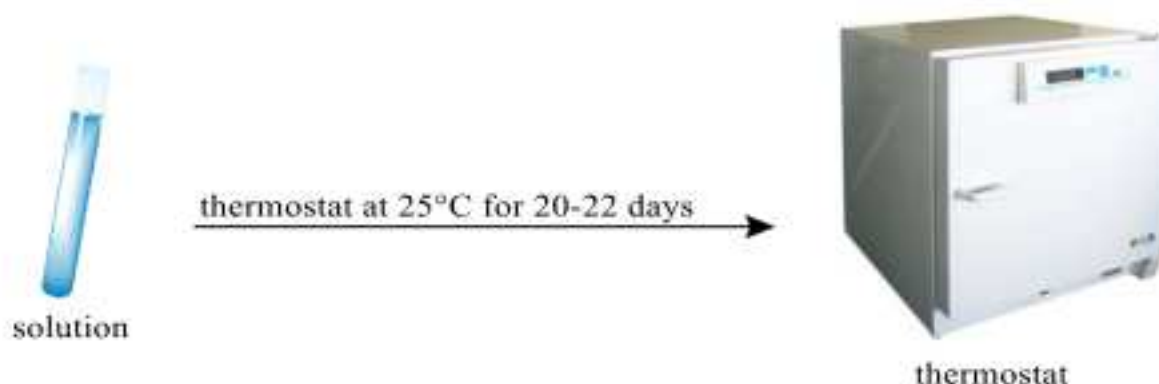
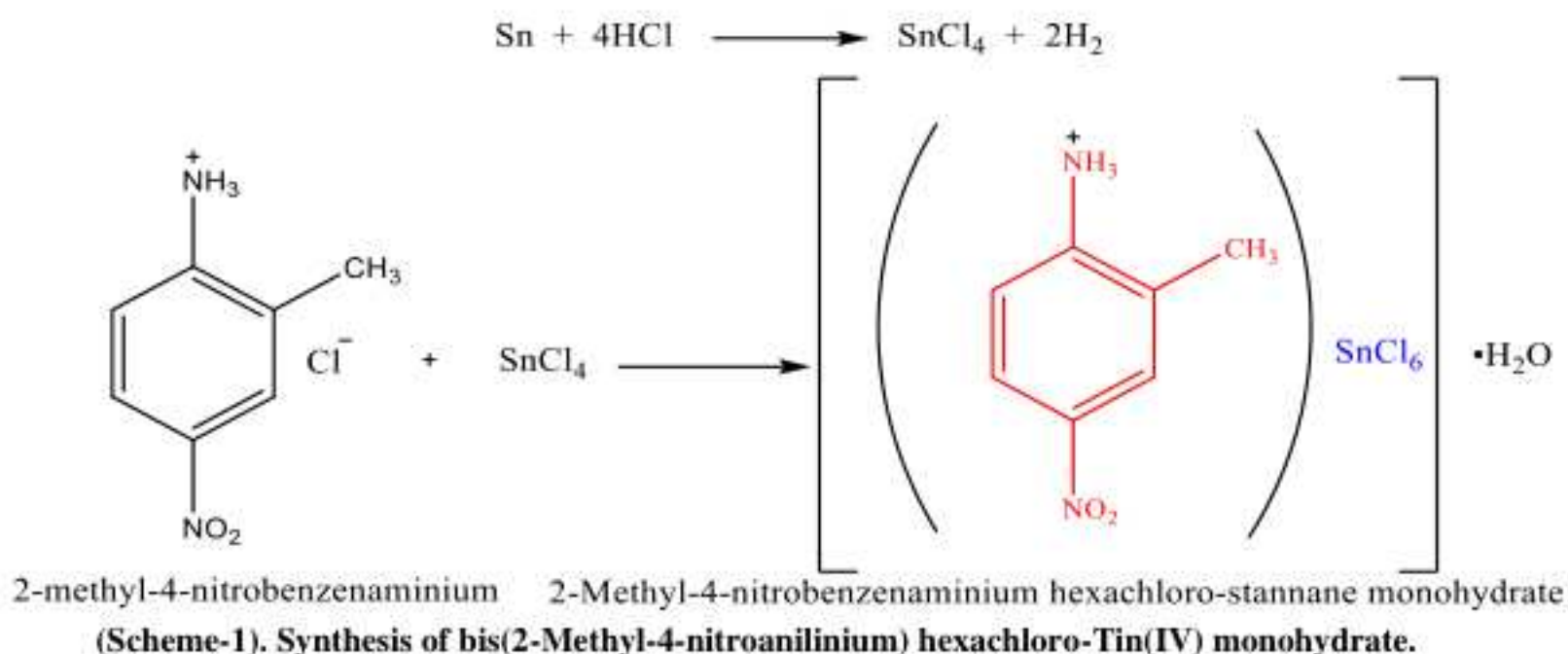
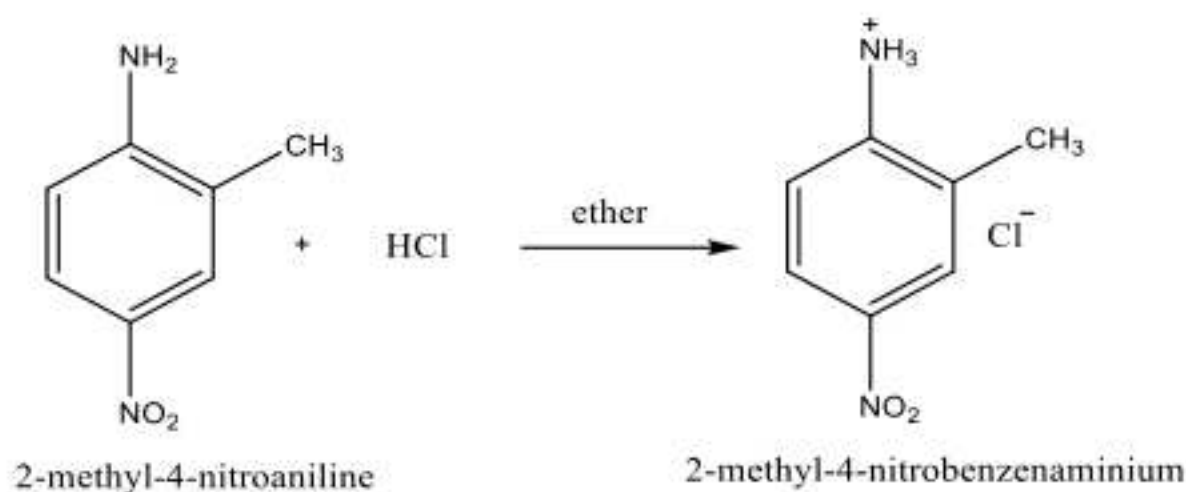
Fig-2. Packing of molecules in the structure of nonlinear optical (NLO) materials. bis(2-Methyl-4-nitroanilinium) hexachloro-Tin(IV) monohydrate

2. Experimental. *Synthesis of bis(2-Methyl-4-nitroanilinium) hexachloro-Tin(IV) monohydrate.*

Synthesis: The initial compounds, 2-Methyl-4-nitroaniline. All reagents and solvents used in the synthesis were of reagent grade and employed without additional purification. Yellow powder 2-Methyl-4-nitroaniline [Aldrich, purum > 98% (NT)], (0.152g 10 mM) and hydrochloric acid (Aldrich, 35 % in H_2O , 99.95%), (2 mL) were mixed in diethyl ether (10 mL) at room temperature to form a 2-Methyl-4-nitroanilinium chloride precursor solution. The solution was heated to 60°C and kept at this temperature for 10 min. Upon

cooling, 2-Methyl-4-nitroanilinium chloride precipitated in the solution. The precipitate was then washed with diethyl ether and dissolved in ethanol. Tin shots, weighing 0.118 g (5 mM), were dissolved in 5 mL of 37 % hydrochloric acid at room temperature over seven hours to produce form **tin chloride** (SnCl_4). The tin chloride solution was added to the freshly prepared ethanolic solution of the 2-Methyl-4-nitroanilinium chloride precursor while continuously stirring (Scheme-1).

The solution was placed in a thermostat at 25°C for 20-22 days, resulting in the formation of a new crystal (Scheme-2).



(Scheme-2). Synthesis of bis(2-Methyl-4-nitroanilinium) hexachloro-Tin(IV) monohydrate.

3. Analysis of Hirshfeld surfaces of discrete nonlinear optical (NLO) materials. Molecular crystalline structures can be quantitatively analyzed through the Hirshfeld surface technique. This methodology illuminates the shifts happening at every point on the Hirshfeld surface, pertaining to neighboring atoms situated on both the external and internal surfaces. These distinct attributes have been pivotal in scrutinizing the selectivity and specificity of intermolecular forces acting upon molecular arrangements[13].

The creation of these surfaces involves partitioning the space enclosed by the crystal using

the Hirshfeld ratio, where the procrystal is effectively delineated by employing a promolecule characterized by an electron density of 0.5. The normalized contact distance, referred to as d_{norm} , is calculated by considering perspectives from both the exterior and interior of the surface, as outlined below:

$$d_{norm} = \frac{d_i - r_i^{vdW}}{r_i^{vdW}} + \frac{d_e - r_e^{vdW}}{r_e^{vdW}}$$

The Hirshfeld surface analysis elucidates the intricate relationships between the external and internal components of a crystalline structure[14]. The parameter denoted as d_{norm} represents the

normalized contact distance, where d_e signifies the distance from the Hirshfeld surface to the nearest external nucleus, d_i denotes the corresponding distance to the nearest internal nucleus, and r^{vdW} designates the van der Waals radius of the atom in question.

The d_{norm} parameter is visually depicted on the Hirshfeld surface through a color gradient ranging from red to white to blue. The bright red regions indicate intermolecular contacts occurring within a distance shorter than the respective van der Waals radii, while the blue regions signify intermolecular contacts at distances greater than the van der Waals radii. The white regions correspond to the cumulative van der Waals radii for the associated atoms [15].

To analyze the short-range contacts between neighboring molecules in crystal lattice structures, the researchers utilized CrystalExplorer version 17 [16]. This software was employed to examine the Hirshfeld surfaces of the crystal structures and generate the corresponding two-dimensional (2D) fingerprint plots [17].

The analysis of Hirshfeld surfaces is an important tool for understanding the intermolecular interactions and crystal packing in nonlinear optical (NLO) materials. Here are some key insights from the search results.

1. The Hirshfeld surface analysis can provide valuable information about the nature and strength of intermolecular interactions that stabilize the crystal structure of NLO materials [18]. This includes hydrogen bonding, π - π stacking, and other noncovalent interactions.

2. The Hirshfeld surface analysis can reveal the contribution of different types of intermolecular contacts (e.g., C-H...Cl; C-H...O; C-H...Br) to the overall crystal packing [3;7]. This information is crucial for understanding the structure-property relationships in NLO crystals.

3. The Hirshfeld surface analysis can also be used to visualize the molecular shape and surface properties of NLO chromophores, which can provide insights into their nonlinear optical response [20]. The shape and charge distribution of the molecules can influence their second-order and third-order nonlinear optical susceptibilities.

4. In addition to Hirshfeld surface analysis, other computational techniques such as density functional theory (DFT) calculations can be employed to further investigate the electronic structure, molecular orbitals, and nonlinear optical properties of NLO materials [17;20]. These complementary analyses can help in the rational design of new NLO materials with improved performance.

5. The search results highlight several examples of NLO crystals, such as stilbazolium derivatives, Schiff base complexes, and organic-inorganic hybrid materials, where Hirshfeld surface analysis has been used to understand the structure-property relationships [7-11].

Hirshfeld surfaces are used in chemistry to analyze intermolecular interactions in crystals, particularly in nonlinear optical materials.

Volume: The volume of a Hirshfeld surface refers to the total space enclosed by the surface, representing the region where intermolecular interactions occur.

Area: The area of a Hirshfeld surface is the total surface area exposed, providing insights into the extent of molecular interaction regions.

Globularity: Globularity is a measure of how spherical or globular a molecule is. In the context of Hirshfeld surfaces, it can indicate the overall shape or compactness of the molecule.

Asphericity: Asphericity is a measure of how much a surface deviates from being perfectly spherical. In Hirshfeld surfaces, it can give information about the molecular shape and symmetry.

Hirshfeld analysis. Hirshfeld surface analysis was performed on a (NLO) compound using its CIF file. The asymmetric part of the structure was selected for the surface analysis.

During the analysis, the Hirshfeld surface was obtained from the normalized contact distance and its d_i and d_e indicators.

Quantitative analysis of the Hirshfeld surface area (d_{norm}) revealed that the total volume is 292.86 Å³ and the surface area is 294.76 Å². Generally, larger volume and surface area indicate more extensive intermolecular bonding and interactions within the crystal structure (Fig-3).

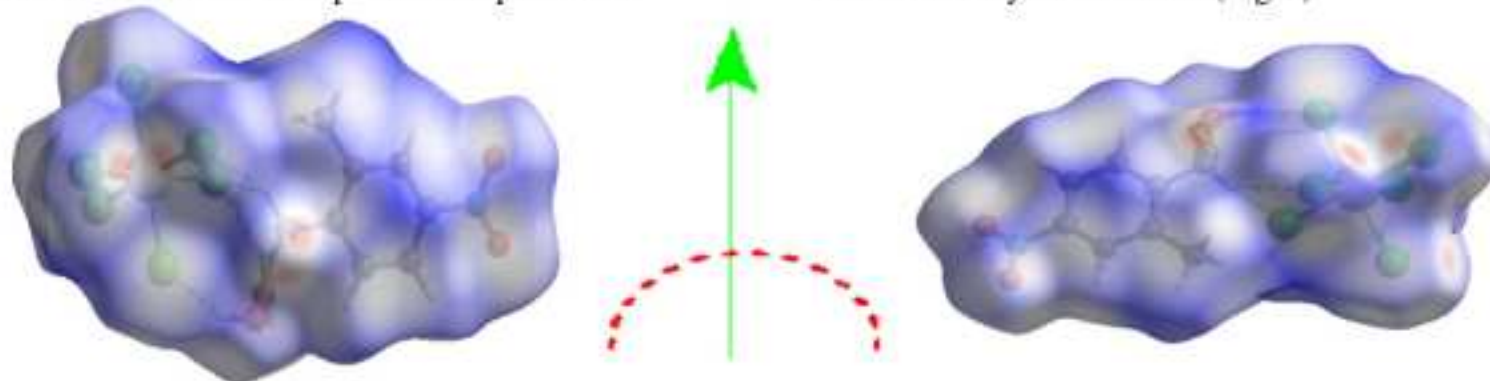


Fig-3. Hirschfeld surface of dnorma nonlinear optical (NLO) materials, interactions inside and outside the surface.

The 2D fingerprints of the Hirshfeld surface of the coordination polymer compound are shown in Fig-5. Analysis of these fingerprint patterns revealed seven different homo- and heteroatomic contact interactions within and between molecules that contribute to the overall crystal packing.

The most prominent intermolecular interactions are H-O/O-H contacts, which account for 55.1% of the Hirshfeld surface. This indicates the important role of hydrogen bonding in stabilizing the crystal structure.

The second largest contribution is H---H contacts, accounting for 11.9% of the surface area.

These close hydrogen-hydrogen interactions also play an important role in intermolecular packing.

Other significant contributions include C---C contacts (1.5%). These reflect the various van der Waals and dipole-dipole interactions present in the crystal.

In slightly smaller proportions, there are All contacts (100%), All---H/H---All contacts (56.9%), O---H/H---O contacts (8.5%), Cl---O/O---Cl (2.6%) and C---H/H---C (2%) and N---O/O---N (0.5%) contacts, all of which also contribute to the Hirshfeld surface. This indicates a variety of intermolecular interactions that stabilize the crystal structure.

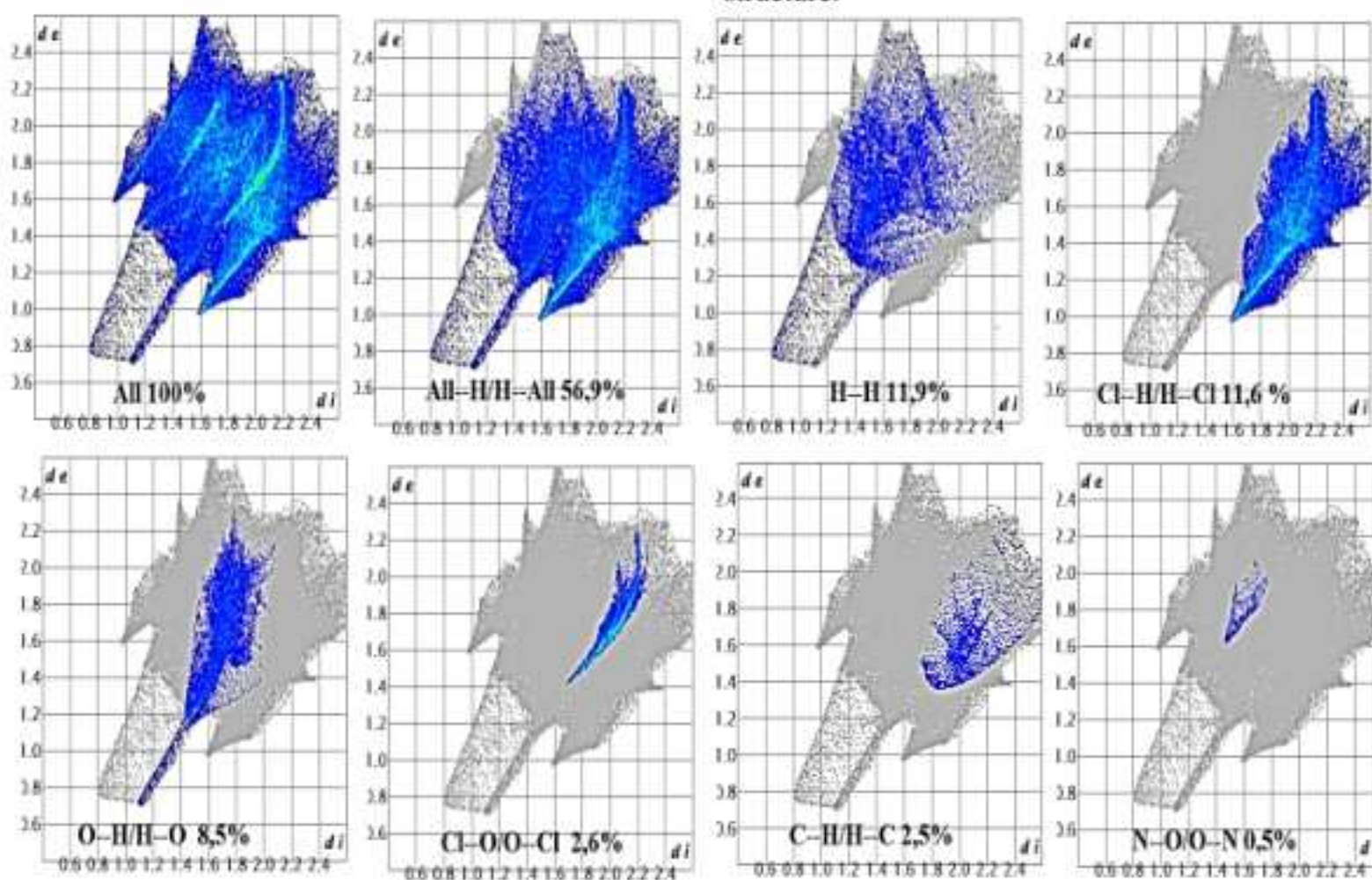


Fig-4. Fingerprint representation of a nonlinear optical (NLO) materials Hirshfeld surface, contribution of interatomic interactions to surface formation

Hydrogen contributes 51.3% to the inner part of the surface and 55.2% to the outer part. Oxygen contributes 36.3% to the inner part and 32.5% to the outer part. Carbon and the tin metal each contribute 0.5% and 0.8% respectively, with the same percentages for both the inner and outer parts of the surface.

This indicates that the contributions of the various elements to the Hirshfeld surface formation are comparable between the inner and outer parts of the molecular surface.

4. Crystal Voids.

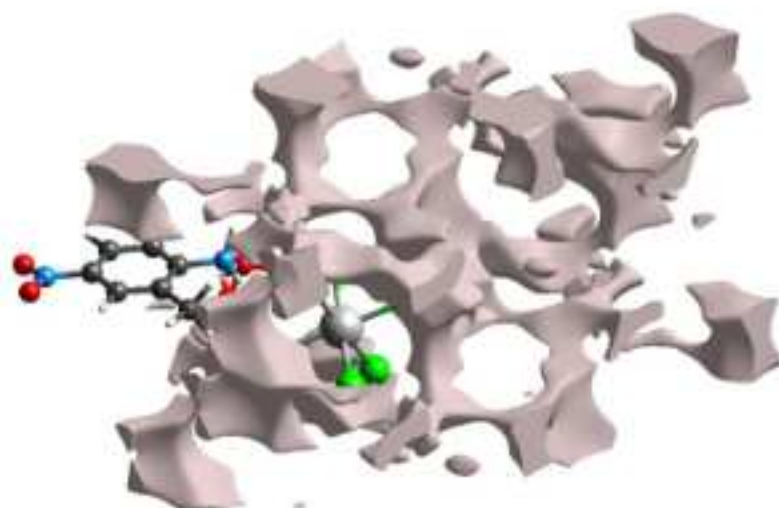


Fig-4. Crystal Voids appearance of bis(2-Methyl-4-nitroanilinium) hexachloro-Tin(IV) monohydrate.

1. Crystal Voids

Volume: 203.45 Å³ – The voids in a crystal represent the empty spaces or cavities within its structure. A void volume of 203.45 Å³ suggests that there's a significant amount of space inside the crystal, potentially affecting its density or porosity.

Area: 743.21 Å² – This is the surface area of the voids within the crystal. A larger surface area in relation to the volume indicates more exposed empty space, which can influence how the crystal interacts with its environment, for example, in adsorption or diffusion processes[21].

2. Globularity: 0.225

Globularity measures how close the shape of an object (or void, in this case) is to being spherical. A value of 1 would mean the void is perfectly spherical, while a value closer to 0 means the shape is more elongated or irregular.

A globularity of 0.225 suggests that the voids in this crystal structure are quite irregular, not close to spherical. This can indicate that the internal structure of the crystal is complex and non-uniform.

3. Asphericity: 0.228

Asphericity measures how much a shape deviates from being a perfect sphere. A value of 0 means the shape is a perfect sphere, and higher values mean the shape is more elongated or distorted.

An asphericity value of 0.228 indicates that the voids are slightly distorted but not extremely elongated. The voids are not perfectly spherical, but their distortion isn't extreme either.

Summary Analysis: The crystal has notable voids, with a reasonably large volume and surface area, which may affect its physical properties such as porosity, adsorption, or interaction with other materials.

The voids are not very globular, meaning their shape is irregular, and the asphericity suggests that they are not perfectly spherical, though they are not highly elongated either.

In essence, the crystal contains irregularly shaped internal voids that could significantly influence its material properties, particularly in fields like catalysis, filtration, or material science, where void spaces play an important role in functionality.

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