

One-dimensional Anion-chain Containing $[\text{Mo}_2\text{O}_6(\text{pic})_2]^{2-}$ Unit Connected by Unclassical Hydrogen Bond

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The reaction of picolinic acid (Hpic) and $(n\text{-Bu}_4\text{N})_4[\text{Mo}_8\text{O}_{26}]$ in the presence of CH_3CN gave a supramolecular compound $(n\text{-Bu}_4\text{N})_2[\text{Mo}_2\text{O}_6(\text{pic})_2]$ (1), and its structure was characterized by elemental analysis, IR spectra and single crystal X-ray diffraction analysis. The single crystal X-ray diffraction study shows that the crystal is in monoclinic crystal system, $P2_1/c$ space group with cell parameters $a = 0.9941(2)$, $b = 1.3719(2)$, $c = 1.8824(2)$ nm, $\beta = 100.840(10)^\circ$, $M_r = 508.50$, $V = 2.5214(9)$ nm³, $F(000) = 1072$. Compound 1 is a dimer through two $\mu_2\text{-O}$ bridging to two $\text{MoO}_3(\text{pic})^-$ unit, then connected one-dimensional anion-chain by unclassical hydrogen bonding, and the anion-chain across the channels formed via $(n\text{-Bu}_4\text{N})^+$ cations. By means of unclassical hydrogen bonding, the anion-chain and cation-channels assemble into three-dimensional supramolecular networks.

Keywords molybdenum(VI) complex, crystal structure, hydrogen bonding, supramolecular networks

Introduction

During the past decades, the development of the coordination chemistry of molybdenum(VI) focused on metal-oxygen clusters characterized by fascinating structural, electrochemical, catalytic, magnetic, medicinal, and photophysical properties,¹ which are of fundamental and practical interest.²⁻⁴ Work on molybdenum compounds has also been stimulated by the metal-metal multiple bonds and supramolecular architecture.⁵⁻⁸ On the other hand, molecular self-assembly has been achieved by hydrogen

bonds between organic components.⁹ Recently, Shriver¹⁰ reported a hydrogen-bonded hexamolybdenum clusters; formation of inorganic-organic networks. An advantage of this approach is that hydrogen bonds are relatively easy to direct. Although several molybdenum complexes of picolinic acid have been investigated,¹¹⁻¹⁵ the crystal structural data and noncovalent bonding of them are absent. Herein we reported a one-dimensional anion-chain containing $[\text{Mo}_2\text{O}_6(\text{pic})_2]^{2-}$ unit which is connected by unclassical hydrogen bonding.

Experimental

Preparation of $(n\text{-Bu}_4\text{N})_2[\text{Mo}_2\text{O}_6(\text{pic})_2]$ (1)

A solution of picolinic acid (Hpic) (0.142 g, 1 mmol) in 10 mL of CH_3CN was added dropwise to a solution of $(n\text{-Bu}_4\text{N})_4[\text{Mo}_8\text{O}_{26}]$ (1.05 g, 0.5 mmol) in CH_3CN (40 mL). The reaction mixture was stirred at room temperature for 5 h to give a clear pale-yellowish solution. This clear solution was allowed to stand for several days to give out pale-yellowish block crystals. Yield 0.110 g (40%); ¹H NMR (DMSO-*d*₆) δ : 8.37—7.22 (Hpic), $[\{\text{C}(\delta)\text{H}_3\text{C}(\gamma)\text{H}_2\text{C}(\beta)\text{H}_2\text{C}(\alpha)_2\}_4\text{N}]^+$: 3.16 (H α), 1.56(H β), 1.30(H γ), 0.93(H δ); IR (KBr) ν : 3481(m), 3285(w), 2960(s), 2936(s), 2874(s), 1704(w), 1651(s), 1600(s), 1571(m), 1489(s), 1468(m), 1446(m), 1382(m), 1354(s), 1295(m),

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1247(m), 1198(m), 1170(m), 1147(m), 1129(w), 1107(w), 1087(m), 1045(m), 1021(m), 977(m), 902(s), 878(s), 854(m), 827(w), 768(s), 754(s), 699(s), 638(m), 573(w), 535(w), 515(w), 453(m), 419(m) cm^{-1} . Anal. calcd for $\text{C}_{22}\text{H}_{40}\text{MoN}_2\text{O}_5$: C 51.93, H 7.86, N, 5.50; found C 51.98, H 7.93, N 5.60.

X-ray crystallography

A single crystal with dimensions 0.60 mm \times 0.50 mm \times 0.40 mm was mounted and data collection were performed on a Siemens-P4 four-circle diffractometer by

ω -scan techniques using graphite-monochromated MoK α radiation ($\lambda = 0.071073$ nm). Details of the crystal parameters, data collection and refinement are listed in Table 1. Atomic coordinates and equivalent isotropic displacement parameters are listed in Table 2. Selected bond distances, angles and hydrogen bond are listed in Table 3 and Table 4. The crystal structure was solved by direct method used SHELXS-86¹⁶ and refined by full-matrix least-square calculations on F^2 used SHELXL-93.¹⁷ All non-hydrogen atoms were refined anisotropically, whereas the hydrogen atoms were generated geometrically. Calculations were performed on a PC computer using Siemens SHELXTL programs package.^{18,19}

Table 1 Crystallographic data for compound 1

Empirical formula	$\text{C}_{22}\text{H}_{40}\text{MoN}_2\text{O}_5$	D_c ($\text{Mg} \cdot \text{m}^{-3}$)	1.340
Formula weight	508.50	μ (mm^{-1})	0.552
T (K)	297(2)	2θ range ($^\circ$)	3.70 to 50.00
Crystal system	Monoclinic	Index range	$0^\circ \leq h \leq 11^\circ$, $0^\circ \leq k \leq 16^\circ$, $-22^\circ \leq l \leq 21^\circ$
Space group	$P2_1/c$	Reflections collected	5100
a (nm)	0.9941(2)	Independent reflections	4413 ($R_{\text{int}} = 0.0173$)
b (nm)	1.3719(2)	Data/restraints/parameters	4413/0/276
c (nm)	1.8824(5)	Goodness-of-fit on F^2	1.029
β ($^\circ$)	100.840(10)	Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0318$, $wR_2 = 0.0848$
V (nm^3), Z	2.5214(9), 4	R indices (all data)	$R_1 = 0.0440$, $wR_2 = 0.0890$
$F(000)$	1072	Largest diff. peak and hole	527 and -383 e/nm^3

$$R = \frac{\sum \|F_o - |F_c|\|}{\sum |F_c|}, wR_2 = \frac{\sum w(|F_o|^2 - |F_c|^2)}{\sum |w(F_o)^2|^{1/2}}, w = 1/[\sigma^2(F_o^2) + (0.0827P)^2], P = (F_o^2 + 2F_c^2)/3.$$

Table 2 Atomic coordinates and equivalent isotropic displacement parameters [$\times 10^4$] for 1

	X	Y	Z	$U(\text{eq})$
Mo	-0.52682(2)	0.407099(18)	0.545642(12)	0.03783(11)
O(1)	-0.4436(2)	0.42446(14)	0.65801(10)	0.0443(5)
O(2)	-0.2690(2)	0.44126(18)	0.74969(11)	0.0630(6)
O(3)	-0.5291(2)	0.43558(14)	0.45176(10)	0.0411(5)
O(4)	-0.5128(2)	0.28297(16)	0.54932(11)	0.0583(6)
O(5)	-0.6875(2)	0.42958(16)	0.56179(12)	0.0551(6)
N(1)	-0.2824(2)	0.40522(17)	0.56461(13)	0.0443(6)
N(2)	0.3963(2)	0.22625(18)	0.77740(13)	0.0457(6)
C(1)	-0.2055(3)	0.3938(2)	0.51365(18)	0.0539(8)
C(2)	-0.0649(4)	0.3974(3)	0.5310(2)	0.0663(10)
C(3)	-0.0009(4)	0.4133(3)	0.6009(2)	0.0694(11)
C(4)	-0.0800(3)	0.4231(2)	0.6532(2)	0.0610(9)
C(5)	-0.2202(3)	0.4188(2)	0.63262(16)	0.0424(7)
C(6)	-0.3149(3)	0.4288(2)	0.68579(16)	0.0442(7)
C(7)	0.4637(3)	0.3194(2)	0.80973(17)	0.0526(8)

	X	Y	Z	U(eq)
C(8)	0.5429(4)	0.3123(3)	0.88649(18)	0.0665(10)
C(9)	0.5890(5)	0.4107(3)	0.9156(2)	0.0868(13)
C(10)	0.6690(6)	0.4077(4)	0.9920(3)	0.1139(19)
C(11)	0.3283(3)	0.2488(2)	0.69976(16)	0.0554(8)
C(12)	0.2527(6)	0.1652(3)	0.6585(2)	0.119(2)
C(13)	0.1862(6)	0.1762(4)	0.5885(3)	0.128(2)
C(14)	0.0907(6)	0.0998(4)	0.5579(4)	0.154(3)
C(15)	0.2919(3)	0.1899(2)	0.82070(17)	0.0530(8)
C(16)	0.1807(4)	0.2612(3)	0.8285(2)	0.0671(10)
C(17)	0.0818(4)	0.2175(4)	0.8713(2)	0.0879(13)
C(18)	-0.0404(5)	0.2847(4)	0.8733(3)	0.1131(18)
C(19)	0.5017(3)	0.1460(2)	0.77927(18)	0.0528(8)
C(20)	0.6165(4)	0.1673(3)	0.7390(2)	0.0637(9)
C(21)	0.7171(5)	0.0835(3)	0.7463(3)	0.0996(15)
C(22)	0.8250(6)	0.0967(4)	0.7015(3)	0.132(2)

Continued

Table 3 Selected bond lengths (nm) and angles (°)

Mo—O(3)	0.18059(19)	Mo—O(1)	0.2135(2)	Mo—O(4)	0.1709(2)
Mo—O(5)	0.1709(2)	Mo—O(3A)	0.2227(2)	Mo—N(1)	0.2388(2)
O(1)—C(6)	0.1289(4)	O(2)—C(6)	0.1216(3)	N(1)—C(5)	0.1326(4)
N(1)—C(1)	0.1344(4)	C(1)—C(2)	0.1375(5)	C(2)—C(3)	0.1368(5)
C(3)—C(4)	0.1377(5)	C(4)—C(5)	0.1376(4)	C(5)—C(6)	0.1504(4)
N(2)—C(7)	0.1516(4)	C(7)—C(8)	0.1513(4)	C(8)—C(9)	0.1495(5)
C(9)—C(10)	0.1508(6)	C(11)—C(12)	0.1504(5)	C(12)—C(13)	0.1366(5)
C(13)—C(14)	0.1457(7)	C(15)—C(16)	0.1504(5)	C(16)—C(17)	0.1507(5)
C(17)—C(18)	0.1530(6)	C(19)—C(20)	0.1512(4)	C(20)—C(21)	0.1513(5)
C(21)—C(22)	0.1495(6)				
O(4)—Mo—O(5)	104.14(11)	O(4)—Mo—O(3)	103.96(9)	O(5)—Mo—O(3)	107.15(10)
O(4)—Mo—O(1)	93.30(9)	O(5)—Mo—O(1)	90.27(9)	O(3)—Mo—O(1)	151.24(8)
O(4)—Mo—O(3A)	161.23(10)	O(5)—Mo—O(3A)	93.37(9)	O(3)—Mo—O(3A)	76.64(8)
O(1)—Mo—O(3A)	79.72(7)	O(4)—Mo—N(1)	84.86(9)	O(5)—Mo—N(1)	159.02(10)
O(3)—Mo—N(1)	88.49(9)	O(1)—Mo—N(1)	70.08(8)	O(3A)—Mo—N(1)	76.38(8)
C(6)—O(1)—Mo	125.26(18)	Mo—O(3)—Mo(A)	103.36(8)	C(5)—N(1)—C(1)	118.8(3)
C(5)—N(1)—Mo	114.78(19)	C(1)—N(1)—Mo	126.4(2)	O(2)—C(6)—O(1)	124.6(3)

Table 4 Selected hydrogen bond (nm)

Donor—H...Acceptor	[ARU]	D...A
C(3)—H(3)...O(5)	[1655.01]	0.3341(5)
C(7)—H(7A)...O(2)	[1655.01]	0.3502(4)
C(10)—H(10A)...O(4)	[4655.01]	0.3465(6)
C(15)—H(15B)...O(3)	[4655.01]	0.3250(4)

1 - x - 1, - y + 1, - z + 1; [1655] = 1 + x, y, z; [4655] = 1 + x, 1/2 - y, 1/2 + z

Spectrum and physical measurements

Elemental analyses were taken on a Perkin-Elmer

240 elemental analyzer. Infrared (IR) spectra were recorded on a Nicolet FT-IR 170SX spectrophotometer using KBr discs. ¹H NMR (500 MHz) spectroscopic measurements were performed on a Bruker AM-500 NMR spectrometer.

Results and discussion*Crystal structure*

The crystal structure of **1** consists of *n*-Bu₄N⁺

cation and $[\text{Mo}_2\text{O}_6(\text{pic})_2]^{2-}$ complex anion (Fig. 1), in which no classical hydrogen bonding interaction exists and is similar to $(\text{Bu}_4\text{N})_2[\text{Mo}_2\text{O}_5(\text{mp})_2]$ ($\text{mp} = o$ -mercaptophenolate).²⁰ The complex anion is a dimer through two μ^2 -O [O(3) and O(3A)] bridging to two identical units containing $\text{MoO}_3(\text{pic})^-$, which shows approximately C_{2v} symmetry. Each molybdenum atom adopts distorted octahedron geometry and shares a common edge with another identical Mo atom to provide a con-edge bi-octahedral structure. The Mo atoms are twice bridged by two O atoms (μ^2 -O), with the Mo \cdots Mo distance 0.31747(6) nm which is longer than that of $[\{\text{MoO}_2(2\text{-SC}_6\text{H}_4\text{O})\}_2(\mu\text{-O})]^{2-}$ (Mo \cdots Mo 0.315 nm),²¹ indicating the absence of a direct metal-metal interaction between two molybdenum atoms.²⁰ The axial sites of distorted octahedron environment O_5N of the Mo(VI) ions are occupied by the terminal O-atom O(4) and the bridging O-atom O(3A) with *trans*-angle O(4)-Mo-O(3A) of $161.23(10)^\circ$, while the equatorial plane is occupied by the other terminal O-atom [O(5)], the bridging O-atom O(3) and two donor atoms O(1) and N(1) from the chelating picolinato ligand. Similar to that in the structure of $[\text{Mo}(\text{NO})_2(2\text{-pic})_2]$,²² the picolinato anion chelates to the Mo atom through its pyridyl N and one of oxygen atoms of carboxyl group to form a stable five-membered ring. But the distance of Mo—N [0.2388(2) nm] is longer than that of Mo—N [0.2164(5) nm] in $[\text{Mo}(\text{NO})_2(2\text{-pic})_2]$.²² This coordinate mode for pic can be observed in many other picolinato complexes, such as $[\text{dmpdaPt}(\text{pic})](\text{pic})$,²³ $[\text{Mn}(\text{Hpic})(\text{pic})\text{Cl}]_2$,²⁴ $[\text{Dy}(\text{pic})_3(\text{H}_2\text{O})_2]_2 \cdot 8\text{H}_2\text{O}$,²⁵ $[\text{V}(\text{DPA})(\text{PA})(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}$,²⁶ $[\text{Cd}(\text{picolinato})(\text{N}_3)]_n$,²⁷ and $\text{Mn}(\text{pic})(\text{N}_3) \cdot (\text{H}_2\text{O})$.²⁸ The two terminal O-groups O(4) and O(5) are in *cis*-configuration with the same Mo—O distance of 0.1709(2) nm, indicating that they are double bond.²⁹ The resulting O = Mo = O angle $104.14(11)^\circ$ is comparable to that of $(\text{Bu}_4\text{N})_2[\text{Mo}_2\text{O}_5(\text{mp})_2]$,²⁰ $[\{\text{MoO}_2(2\text{-SC}_6\text{H}_4\text{O})\}_2(\mu\text{-O})]^{2-}$,²¹ and $\text{K}_4[(\text{MoO}_2)\text{O}(\text{Hcit})_2] \cdot 4\text{H}_2\text{O}$,²⁹ which is considerably larger than the 90° regular octahedron value for *cis* groups; this is expected from the greater O \cdots O repulsion between oxygen atoms with short bonds to the metal atom. The significantly long Mo—O(3A) distance [0.2227(2) nm] of compound 1 shows weak coordination of the bridging oxygen to molybdenum, which is notable. It suggests that the bridging oxygen is probably much easier to break by another ligand and give

rise to an active catalyst for the oxidation of organic substrates³⁰⁻³² or acting as an oxo transfer agent.³³ In addition, it must be pointed out that the thermal parameters of carbon of ethyl group in cations of compound 1 are higher than those of normal situation because of statistical distribution, which are normal and can be seen in other occasions.²¹

The most important feature of compound 1 is that the unclassical hydrogen bonding exists. As showed in Figs. 2 and 3, the $[\text{Mo}_2\text{O}_6(\text{pic})_2]^{2-}$ unit through unclassical hydrogen bonding³⁴⁻³⁶ [C(3)—H(3) \cdots O(5) 0.3341(5) nm] connects to other identical unit and gives a one-dimension anion-chain (Fig. 4), and the anion-chain across the cation-channels (Fig. 5) formed by $(n\text{-Bu}_4\text{N})^+$. The anion-chain and cation-channels via unclassical hydrogen bonding {C(7)—H(7A) \cdots O(2) [0.3502(4) nm]; C(10)—H(10A) \cdots O(4) [0.3465(6) nm]; C(15)—H(15B) \cdots O(3) [0.3250(4) nm]} assembled to supramolecular three-dimension networks (Fig. 6). The discrete molecules spontaneously self-assemble into extended arrays with channel may has potential application in catalyst.¹⁰

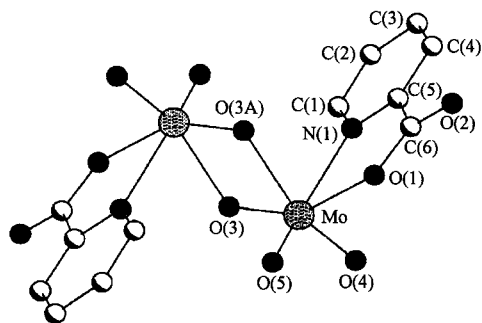


Fig. 1 Crystal structure of the anion in compound 1.

IR spectrum

Compared to the free picolinic acid, the complex shows a slightly decrease of the $\nu_{\text{as}}(\text{COO})$ to 1651 cm^{-1} and a consequent increase of the $\nu_{\text{s}}(\text{COO})$ around 1354 cm^{-1} , which are characteristic of unidentate carboxyl group.^{13,24,27,28} The vibrations related to the pyridine moiety spread over all the spectra are broad, similar to those of the pyridine itself and slightly shifted to higher wave numbers in the solid complex. In the region between 1500 cm^{-1} and 600 cm^{-1} , the complex shows several bands,

which might result from the presence of *cis*-dioxo core and bridging oxo, whereas the $\nu_{as}(O=Mo=O)$ and $\nu_s(O=Mo=O)$ stretch vibrations appear at 902 cm^{-1} and 878 cm^{-1} , which is the characteristic of the *cis*- MoO_2^{2+} group,^{20,21} and it is different from the situation in $[MoO_2 \cdot SAE]_2$.³⁷ While the vibrations of 573 cm^{-1} and 419 cm^{-1} may be assigned to $\nu(Mo-O)$ and $\nu(Mo-N)$ stretches, respectively.³⁸

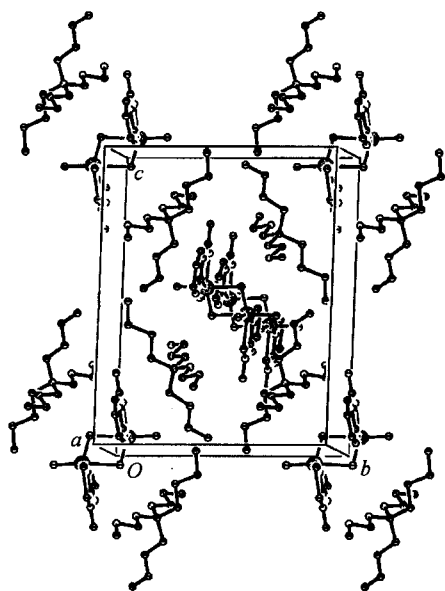


Fig. 2 Molecular packing of compound 1.

1H NMR spectrum

In contrast to the 1H NMR spectrum of picolinic acid, it is found that the multiple peaks for picolinato anion appear in the region of δ 8.37 to δ 7.22, which shifts to low field due to formation of complex. Additionally, the aliphatic protons belong to $n-Bu_4N^+$ may be assigned to $[C(\delta)H_3C(\gamma)H_2C(\beta)H_2C(\alpha)_2]_4N^+$: 3.16 (H α), 1.56 (H β), 1.30 (H γ), 0.93 (H δ).²¹

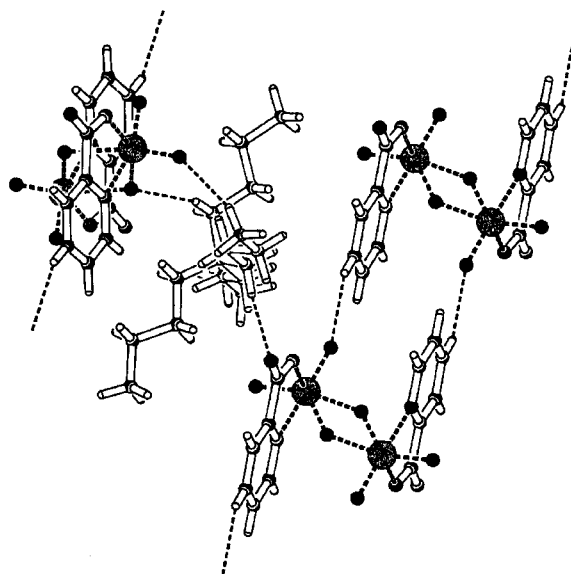


Fig. 3 Hydrogen bonding interaction for compound 1.

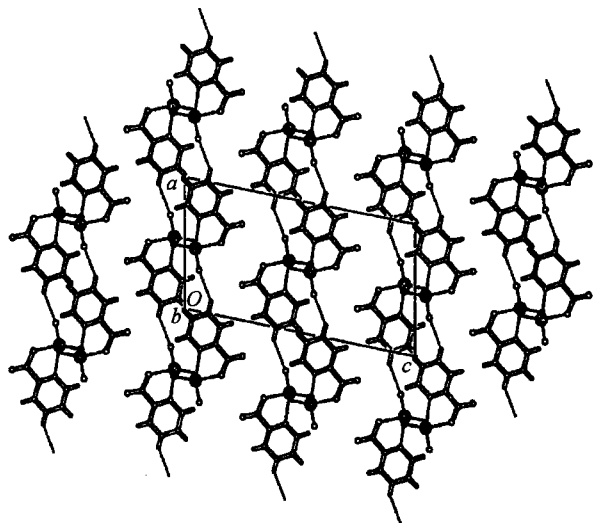


Fig. 4 One-dimensional anion-chain form by $[Mo_2O_6(pic)_2]^{2-}$

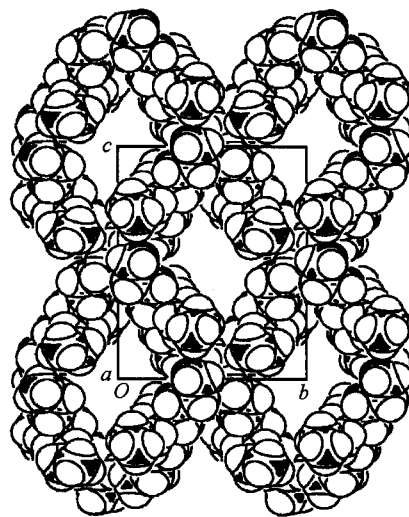


Fig. 5 Cation-channel constituted by $(n-Bu_4N)^+$.

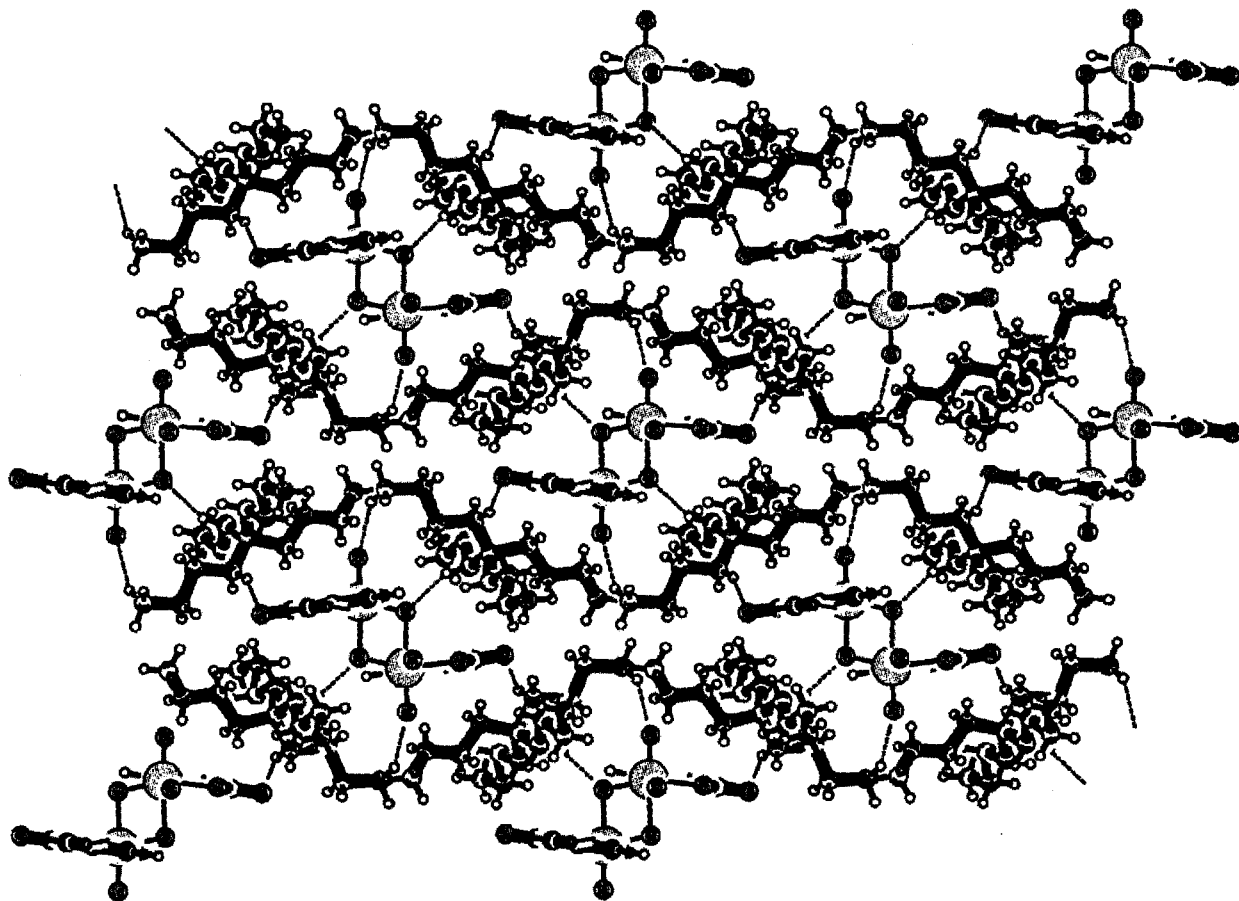


Fig. 6 Supramolecular three-dimensional networks for compound 1.

Supplementary material

Supplementary materials of the crystal determination for compound 1 have been deposited with the Cambridge Crystallographic Data Center as Supplementary Publication no. CCDC: 158002.

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