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Synthesis and characterization of the adducts of bis(O-butylxanthato) Ni(II) with nitrogen donor ligands and X-ray structure of bis(Obutylxanthato)bis(3-chloropyridine)nickel(II)



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ABSTRACT

A new series of adducts of bis(*O*-butylxanthato)nickel(II) with substituted pyridines have been synthesized by treating bis(*O*-butylxanthato)nickel(II) with substituted pyridines in acetone. The complexes have been characterized by elemental analysis, conductivity measurements, magnetic susceptibility measurements, TGA/DTA studies, infrared and electronic spectral studies. X-ray studies of one of the adduct bis(*O*-butylxanthato)bis(3-chloropyridine)nickel(II) shows that the central metal is octahedrally coordinated within a *trans*-N₂S₄ donor set, with the Ni atom located on a centre of inversion. The complex crystallizes in the monoclinic space group P2₁/c with unit cell parameters *a* = 11.8088(8) Å, *b* = 12.2042(7) Å, *c* = 9.0252(5) Å, β = 93.789(6), *Z* = 2. Crystal structure was solved by direct methods and refined by full matrix least squares procedures to a final R-value of 0.0380 (*w*R2 = 0.0885) for 2083 observed reflections. The butyl chain is disordered over two set of sites, with occupancy ratios of 0.741:0.259. These studies suggest a distorted octahedral structure and paramagnetic nature of the adducts.

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1. Introduction

In recent few years there has been considerable upsurge of knowledge in the study of chemistry of an important class of bidentate sulfur donating ligands, called xanthates, a name derived from the Greek word xanthos (yellow), owing to the yellow color of lead xanthates [1,2]. Xanthates as a ligand are widely investigated due to their diverse coordination behavior [3,4]. They can form bidentate, monodentate or network solids, showing a wide range of coordination behavior [5–7]. Metal xanthates are extensively used as corrosion inhibitors and agricultural reagents [8–10]. Xanthates have also found important use in medicine as antitumor agents and for treating Alzheimer's disease [11–13]. Sodium and potassium ethylxanthate have antidotal effects in acute mercurial poisoning and recently as co-adjuvant in AIDS treatment [14,15].

Interestingly, there are no significant reports of xanthate ligand functionalized with additional donor groups that would expand their coordination capabilities but some how, by modifying the

* Corresponding author. E-mail address: rkant.ju@gmail.com (R. Kant). backbone of these ligands with a second donor set (for instance with hard donors such as pyridine-based groups), it is possible to take advantage of their rich coordination patterns and differences between the coordination preferences of sulfur atoms for soft metals and of pyridine-based groups for hard metals to design and prepare new heterometallic species. Thus in continuation of our earlier research work on alkylxanthates and their adducts with nitrogen donor ligands, we herein report the synthesis and characterization of adducts of bis(O-butylxanthato)nickel(II) with substituted pyridines.

2. Experimental section

2.1. Synthesis of the complex

2.1.1. Preparation of potassium salt of O-butylxanthate

The potassium salt of *O*-butylxanthate was prepared by the standard published method [16]. Into a 500 ml round bottomed flask, fitted with a reflux condenser, 4.2 g (0.075 mol) of potassium hydroxide pellets were placed and 19.271 g (23.472 ml, 0.26 mol) of n-butanol was added. The reaction mixture was heated under reflux for 1 h. The contents were then cooled and the liquid from

Table 1

Analytical data, molar conductance and magnetic moments of the adducts of bis(O-butyldithiocarbonato)nickel(II) with substitute	d pyridines
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S. No	Name of the adduct	%Found(calculate)				Magnetic moment $\mu_{eff}(B.M)$	Molar conductance ($Ohm^{-1} mol^{-1} cm^2$)
		С	Н	Ν	S		
1	Bis(O-butylxanthato) (2-ethylpyridine)nickel(II)	48.95 (50.28)	5.88 (6.28)	4.35 (4.88)	22.05 (22.35)	2.93	50.25
2	Bis(O-butylxanthato) (3-ethylpyridine)nickel(II)	49.01 (50.28)	5.93 (6.28)	4.05 (4.18)	22.0 (21.85)	3.48	40.72
3	Bis(O-butylxanthato) (4-ethylpyridine)nickel(II)	48.82 (50.28)	6.01 (6.28)	4.43 (4.63)	21.95 (22.47)	3.19	48.39
4	Bis(O-butylxanthato) (2-chloropyridine)nickel(II)	39.53 (41.04)	4.2 (4.4)	4.48 (4.54)	20.93 (21.89)	3.02	39.24
5	Bis(O-butylxanthato) (3-chloropyridine)nickel(II)	39.98 (41.04)	4.01 (4.4)	4.23 (4.58)	20.98 (21.44)	3.23	42.12

Table 2

Infrared and UV-Visible Spectra of (O-butyldithiocarbonato)nickel(II) with substituted pyridines.

Name of the adduct	IR spectral data(cm ⁻¹)		UV–visible (cm ⁻¹)			
	vas(C-O-C)	υ _s (C-O-S)	υ(C-S)	$\upsilon_1 \ ^3A_2g \ \rightarrow \ ^3T_2g(F)$	$\upsilon_2 \ ^3A_2g(F) \rightarrow \ ^3T_1g(F)$	$\upsilon_3 {}^3A_2g(F \to) {}^3T_1g(P)$
Bis(O-butyldithiocarbonato) bis(2-ethylpyridine)nickel(II)	1202	1123	1033	12478	19200	23800
Bis(O-butyldithiocarbonato) bis(3-ethylpyridine)nickel(II)	1199	1134	1035	12598	18224	25300
Bis (O-butyldithiocarbonato) bis(4-ethylpyridine)nickel(II)	1199	1140	1037	13668	19990	24520
Bis(O-butyldithiocarbonato) bis(2-chloropyridine)nickel(II)	1200	1137	1037	13950	18890	26400
Bis(O-butyldithiocarbonato) bis(3-chloropyridine)nickel(II)	1210	1135	1034	13780	18800	25840





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Fig. 1. IR spectra of bis(O-butylxanthato)bis(4-ethylpyridine).

the reaction mixture was decanted off into another dry 500 ml flask. To this flask, 5.7 g (4.5 ml, 0.075 mol) of carbon disulphide was added slowly with constant heating.

The contents of the flask were filtered (after cooling in ice) on a sintered glass funnel at the pump and washed with three 25 ml portions of ether. The resulting product potassium *O*-butylxanthate was dried in a vacuum desiccator over anhydrous calcium chloride.

The saturated aqueous solutions of NiCl₂· $6H_2O(2.37 \text{ g}, 0.01 \text{ mol})$ and potassium O-butyldithiocarbonate (3.7 g, 0.02 mol) were prepared separately and then mixed with constant stirring. Dark green precipitates were formed which were filtered immediately and were dried in a vacuum desiccator over anhydrous calcium chloride. The composition of the complex was established to be [Ni(S₂COC₄H₉)₂] by the elemental analysis.

$$ROH + CS_2 + MOH \xrightarrow{Reflux} ROCSS^{-1} + H_2O$$

where, $R = -C_4H_9$ and $M^+ = Na$ or K.

$$(aq)C_4H_9CO \longrightarrow C_4H_9CO \longrightarrow C_4H_$$

S.No	Addition complex	Sample mass (mg)	Residual mass (mg)	Mass loss%		Dec.temp.(°C)	Residue compd.	Residu ~800 °C	al% C
				100-200 °C obs.(calc.)	200-500 °C obs.(calc.)			Found	Calc.
1.	$Ni(S_2COC_4H_9)_2(C_7H_9N)_2$	8.5 g	2.45	59.6 (60.1)	73.8 (75.0)	810 °C	NiS	92.8	92.5
2.	$Ni(S_2COC_4H_9)_2(C_5H_4N\cdot Cl)_2$	10 g	3.51	58.9 (60.5)	76.5 (74.5)	800 °C	NiS	91.9	92.0

 Table 3

 Thermal degradation of some adducts of bis(O-alkylxanthato)nickel(II) with substituted pyridine.

2.1.3. 2.1.3Preparation of adducts of Bis(O-butyldithiocarbonato) nickel(II)with substituted pyridines.

The 1:2 adducts of bis(dithiocarbonato)nickel(II)with substituted pyridines were prepared by stirring bis(*O*-butyldithiocarbonato)nickel(II) (0.0026 mol) in about 70 ml of acetone for about 20 min followed by the addition of substituted pyridines [2-, ,3-,4-ethylpyridine and 2-, 3- chloropyridines] (0.0052 mol) for 20 min. The contents of the reaction mixture were then filtered and allowed to stand for 20–24 h, depositing shining green crystalline solids which were then washed with the solvent used in their preparation and dried over calcium chloride at room temperature. The adducts thus obtained were recrystallised from acetone.



Where X=2-,3-,4-ethyl, 2-,3-chloropyridine

.where X = 2-,3-,4-ethyl, 2-,3-chloropyridine.

2.2. Method

Molar conductance of the adducts was determined on their



Fig. 2. TGA of bis(O-butylxanthato)bis(4-ethylpyridine)Ni(II).

millimolar solution in dimethylformamide by using Digital conductivity meter "Century CC 601". Magnetic measurements were carried out at room temperature by Gouy's method. The infrared spectra of the complexes over the region 4000–400 cm⁻¹ have been recorded using KBr pellets on Perkin Elemer FT-IR Spectrophotometer. The electronic spectra of the adducts were recorded in dimethylformamide in the range 12500 cm⁻¹ to 40000 cm⁻¹ on Systronics 119UV–Visible spectrophotometer. The analytical data, molar conductance and magnetic moments of the adducts isolated are presented in Table 1. Important IR bands and Electronic spectral data are cited in Table 2.

2.3. X-ray data collection and structure refinement

A green block-shaped single crystal of the title compound, measuring 0.30 \times 0.20 \times 0.10 mm, was picked up for X-ray intensity data collection. X-ray intensity data were collected by using an X'calibur Oxford Diffraction system with graphite monochromatic Mo K_{α} radiation ($\lambda = 0.71073$ Å), and reduced with CrysAlis RED [17]. The cell dimensions were determined by the least-squares fit of angular settings of 1915 reflections in the θ range 3.76°–29.05°. A total number of 4674 reflections were collected of which 2541 reflections were unique and 2083 reflections were treated as observed $(I > 2\sigma(I))$. Data were corrected for Lorentz, polarization and absorption factors. The structure was solved by direct methods using SHELXS97 and refined by SHELXL97 [18]. The geometry of the molecule is determined by PLATON [19] and PARST [20] software. All H atoms were geometrically fixed and allowed to ride on their parent C atoms, with C–H distances of 0.93–0.97 Å and with 1.5Ueg (methyl C) and $U_{iso}(H) = 1.2U_{eq}(C)$ for other hydrogen atoms. The final refinement cycle yielded an R-factor of 0.0380 [$wR(F^2) = 0.0885$] for the observed data. The crystallographic data are summarized in Table 5. An ORTEP view of the metal complex is shown in Fig. 4.



Fig. 3. TGA of bis(O-butylxanthato)bis(3-chloropyridine)Ni(II).

Table 4

Antifungal activities of some adducts of bis(O-butyldithiocarbonato)nickel(II) with nitrogen donor ligands against Rhizopus nigricans.

S·No	Adittion complex	Colony diameter (mm)	Concentration	Colony diameter(mm)	%Inhibition I = [(CT)/C] \times 100
1	$Ni(S_2COC_4H_9)_2(C_7H_9N)_2$	80	50	72.8	9.0
			100	55.5	30.6
			150	30.0	62.5
			200	12.6	84.25
2	$Ni(S_2COC_4H_9)_2(C_5H_4N \cdot Cl)_2$	80	50	78.0	2.5
			100	62.7	21.6
			150	40.1	49.87
			200	20.3	74.62

3. Results and discussion

3.1. Preliminary investigations

The adducts of Bis(O-butylxanthato)nickel(II) with substituted pyridines are green coloured crystalline solids soluble in common organic solvents. The molar conductivity values calculated (in the range of 34.4–53.08 ohm⁻¹mol⁻¹cm²) from the conductivities measured on millimolar solutions of the adducts in DMF, support the neutral and non-ionic nature of complexes [21,22]. The magnetic moment values of the adducts prepared vary in the range of 2.98–3.01 BMBM which is in agreement with magnetic moment values observed for octahedral complexes of nickel(II) [23,24]. The assignment of octahedral geometries is also supported by the electronic absorption spectra where absorption maxima of d-d transition shows three bands v^1 , v^2 , v^3 respectively at 14000-16000 cm⁻¹ [${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{2g}(F)$] and 25000–28000 cm⁻¹ [${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{2g}(F)$] and adducts with those of the newly prepared adducts with those of the corresponding free ligands reveals that most of the absorption bands

Table 5

Crystal and experimental data.

CCDC No	980793
Crystal description	Block shaped, Green
Empirical formula	C ₂₀ H ₂₆ C ₁₂ N ₂ Ni O ₂ S ₄
Formula weight	584.28
Melting point	95 °C
Measurement	X'Calibur System
Radiation, wavelength	MoK _α , 0.71073 Å
Unit cell dimensions	a = 11.8088(8)Å, $b = 12.2042(7)$ Å, $c = 9.0252(5)$ Å,
	$eta=93.789(6)^\circ$
Crystal system	Monoclinic
Space group	P2 ₁ /c
Unit cell volume	1297.84(14) Å ³
Density (calculated)	1.370 g/cm ³
Temperature	293(2) K
Absorption coefficient	1.295 mm^{-1}
F (000)	604
Scan mode	ω scan
θ range for entire data collection	$3.76^{\circ} < \theta < 26.00^{\circ}$
Range of indices	−10 < h < 14, −8 < k < 15, −8 < l < 11
Reflections collected/	4674/2541
unique	
R _{int}	0.0304
R _{sigma}	0.0451
Structure determination	Direct Methods
Refinement	Full Matrix Least Squares on F ²
Weighing scheme	$[I \sigma^2 (Fo^2) + (0.0445P)^2 + 0.3917P]$ where
	$P = (Fo^2 + 2Fc^2)/3$
No. of parameters refined	182
Final R	0.0380
$wR(F^2)$	0.0885
Goodness-of-fit	1.057
$(\Delta/\sigma)_{max}$	0.001
Final residual electron	$-0.431 < \Delta ho < 0.473 ~ { m \AA}^{-3}$
density	

of free ligands are shifted in their respective complexes. The newly prepared adducts show two bands varying in the range of 1240–1270 cm⁻¹ and1128–1144 cm⁻¹ which are assigned to $v_{as}(-C-O-C)$ and $v_{s}(C-O-C)$ stretching vibrations. The appearance of a single band for v(C-S) vibration and its position indicate symmetrical bidentate binding of xanthate moiety. The co-ordination of metal atom with nitrogen donor ligand is supported by characteristic red shift observed for the C–H out of plane deformation band at 908 cm⁻¹ (Fig. 1).

3.2. Thermal studies

TGA/DTA studies of the complexes were recorded in the temperature range 30–1000 °C at a heating rate of 10 °C/min in nitrogen atmosphere. The results of thermal analysis of two representing adducts bis(O-butylxanthato)bis(4-ethylpyridine) Ni(II) and bis(O-butylxanthato)bis(3-chloro-pyridine)Ni(II) are summarized in Table 3 (Fig. 2 and Fig. 3). The TG curves show stepwise decomposition processes for these complexes resulting in the formation of NiS as a stable end product as indicated by the following general equations:

TGA of bis(O-butylxanthato) (4-ethylpyridine)nickel(II)

$$\begin{bmatrix} Ni(S_2COC_4H_9)_2(4-ethylpyidine)_2 \end{bmatrix} \longrightarrow \begin{array}{c} 100-200^\circ C\\ -2molecules \ of \ 4-ethylpyridine \\ \begin{bmatrix} Ni(S_2COC_4H_9)_2 \end{bmatrix}^{200-500^\circ C} NiSO_4 \xrightarrow{810^\circ C} NiS \\ TGA \ of \ bis(O-butylxanthato)bis(3-chloropyridine)nickel(II) \\ \end{bmatrix}$$

$$\begin{bmatrix} Ni(S_2COC_4H_9)_2(4-chloropyidine)_2 \end{bmatrix} \longrightarrow \begin{array}{c} 100-200^{\circ}C \\ -2molecules \text{ of } 4-chloropyidine} \\ \begin{bmatrix} Ni(S_2COC_4H_9)_2 \end{bmatrix}^{200-500^{\circ}C} NiSO_4 \xrightarrow{810^{\circ}C} NiS \end{bmatrix}$$

 Bond lengths (Å) and bond angles (°) for non hydrogen atoms (e.s.d's in parenthesis).

 Bond lengths (Å)

 Ni1-N1
 2.115(2)

 Ni1-S2
 2.4395(7)

Ni1–N1	2.115(2)	Ni1 –S2	2.4395(7)
Ni1-S1	2.4550(7)	S2 –C1	1.694(3)
S1-C1	1.680(3)	Cl1 –C9	1.730(3)
01–C1	1.326(3)	01 –C2′	1.43(4)
01–C2	1.459(17)	N1- C6	1.328(3)
N1-C10	1.335(3)		
Bond Angles (°)			
N1-Ni1-S2	89.56(6)	N1-Ni1-S1	89.66(6)
S2-Ni1-S1	73.63(2)	C1-S2-Ni1	82.84(10)
C1-S1-Ni1	82.64(9)	C1-01-C2'	115.4(9)
C1-01-C2	120.0(4)	C6-N1-C10	117.9(2)
C6-N1-Ni1	121.12(19)	C10-N1-Ni1	120.91(18)
N1-C6-C7	122.9(3)	N1-C10-C9	121.8(3)
01-C1-S1	116.5(2)	01-C1-S2	122.7(2)
S1-C1-S2	120.78(16)	01-C2-C3	107.9(9)
C3-C4-C5	110.7(6)	01-C2'-C3'	100(2)



Fig. 4. ORTEP view of the molecule with displacement ellipsoids drawn at 40% probability level. H atoms are shown as small spheres of arbitrary radii.

3.3. Biological studies

The antifungal activity of the parent compound and some of the adducts was tested by Poisoned Food Technique against the pathogenic fungus, *Rhizopus nigricans*. The test solutions were prepared by dissolving the compounds in dimethylsulfoxide. The test solutions

were mixed in the PDA and poured in the petriplates in sterilized conditions inside the Laminar flow. After solidification, the plates were inoculated with seven days old culture of pathogen, *Rhizopus nigricans* by placing 2 mm bit in the centre of the plates. The inoculated plates were incubated at 27 °C for 4 days. The linear growth of fungus in controlled manner was recorded at different



Fig. 5. Packing diagram viewed down the b-axis.

 Table 7

 The Geometry of intramolecular interactions.

D–H A	D-H (Å)	H A (Å)	D A (Å)	$\theta \left[D - H \dots A \left(^{\circ} ight) ight]$
C4'-H3B…01 C2'-H2'2 S2 C4A-H3'1 01	0.966(16) 0.97(23) 1.008(6)	2.506(2) 2.402(1) 2.488(2)	3.016(17) 2.875(3) 2.989(6)	112.9(9) 109.56(5) 110.11(3)

concentrations of the complexes. The antifungal screening data are given in Table 4, which shows that the colony diameter of the fungus decreases as the concentration of the complex increases; that is, all the complexes inhibited the growth of fungus significantly. This shows a linear relationship between concentration and percent inhibition. The increase in antifungal activity is due to faster diffusion of metal complexes as a whole through the cell membrane or due to combined activity effect of the metal and the ligand. Such increased activity of the metal complexes can be explained on the basis of Overtone'sconcept [27] and Tweedy's chelation theory [28]. On chelation, the polarity of the metal ion will be reduced to a greater extent due to overlap of the ligand orbital and partial sharing of the positive charge of the metal ion with donor group.

3.4. X-ray crystallographic analysis

The Ni atom in the title complex is octahedrally coordinated within a trans-N2S4 donor set, with the Ni atom located on a centre of inversion. The Ni(II) atom is coordinated by a distorted octahedral arrangement of four sulfur atom from two chelating xanthate ligands and two nitrogen atoms from two chloropyridines ligands. Bond lengths and bond angles for non-hydrogen atoms are listed in Table 6. The Ni1–N1 bond length is 2.115(2) Å. The Ni–S bond lengths involving the xanthate ligands are 2.455(7) Å for Ni1–S1 and 2.439(7) Å for Ni1–S2. The bond angles in the pyridine ring vary from 112.9(3) to 121.8(3), the average value being 118.3(2); these values of the bond angles and bond distances are in good agreement with those reported for other analogous Ni-dithiocarbonato complexes [29–32]. The C–S bond lengths involving the xanthate ligands are: C1-S1 = 1.680(3) and C1-S2 = 1.694(3) Å. These values are the same and show a double bond character due to delocalization over the two C-S bonds [33]. The plane formed by two sulfur, a nickel and a carbon atoms(Ni1, S1, C1, S2) is planer, and the dihedral angle between this plane and the plane of the chloropyridine ring is 85.87(6), which means that both of these planes are held almost perpendicular to each other. The O1–C1 bond distance [1.326(3) Å] is shorter than O1-C2 and O1-C2' [1.46(2) Å and 1.43 Å], due to hybridization of the carbon atom. The short value of the bond distance for O1-C1 is consistent with a significant contribution of the resonance form of the xanthate anion, which features a formal C=O and a negative charge on each S atom [34]. The butyl chain attached to the dithiocarbonato group contains disorder over two sets of sites with occupancy ratios of 0.741: 0.259. The packing of the molecule within the unit cell, viewed down the b-axis, is shown in Fig. 5. Molecules in the crystal are packed together to form parallel layers. The complex is stabilized by weak intramolecular interactions (Table 7).

4. Conclusions

Analytical results shows that the adducts have 1:2 stoichiometry with general formula $[Ni(S_2COR)_2.2L]$ (L = 2-, 3-,4- ethylpyridines, and 2-, 3- chloropyridines). Thermal studies indicate a three step loss of weight with the formation of NiS as stable end product. Antifungal activity of some complexes has been carried out against the fungal strain *Rhizopus nigricans*. The butyl chain is disordered over two set of sites, with occupancy ratios of 0.741: 0.259. These studies suggest a distorted octahedral structure and paramagnetic nature of the adducts. Very few C–H..O and C–H…S weak intramolecular interactions help stabilize the structure in the unit cell.

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Appendix A

CCDC No . 980793 contain the supplementary crystallographic data for the compound. The data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif by e-mailing data_request @ ccdc.cam.ac.uk, or by contacting The Cambridge Crystallography Data Centre, 12 Union Road, Cambridge, CB2 IEZ, UK. Fax: +44(0) 1223-33603.

References

- [1] E.R.T. Tiekink, I. Haiduc, Prog. Inorg. Chem. 54 (2005) 127-319.
- [2] M. Wang, Q. Zhang, W. Hao, Z.X. Sun, Chem. Cent. J. 5 (2011) 73.
- [3] C.I. Haiduc, D.B. Sowerby, S.F. Lu, Polyhedron 14 (1995) 3389.
- [4] L. Ballester, A. Gutierrez, M.F. Perpinan, Polyhedron 13 (1996) 1103.
- [5] R.G. Xiang, Y. Zh, C.M. Liu, X.Z. You, Polyhedron 16 (1997) 266.
- [6] Z. Travnicek, R. Pastorek, Z. Sindelar, R. Klickar, J. Marek, Polyhedron 14 (1995) 3627.
- [7] R.W. Gable, B.F. Hoskins, G. Winter, Inorg. Chim. Acta 96 (1985) 151.
- [8] Q. Han, J. Chen, X. Yang, L. Lu, X. Wang, J. Phy. Chem. C 111 (2007) 14072.
- [9] D.E. Zelmon, Z. Gebeyehu, D. Tomlin, T.M. Copper, Mat. Res. Soc. Sym. Proc. 519 (1998) 395.
- [10] M. Scendo, Corr. Sci. 47 (2005) 1738.
- [11] W.M. Doane, B.S. Shasha, C.R. Russel, Cont. Rel. Pest 53 (1977) 74.
- [12] W.J. Orts, R.E. Sojka, G.M. Glenn, Agro Food Ind. 37 (2002) 1078.
- [13] A.C. Larsson, S. Oberg, J. Phy. Chem. A 115 (2011) 1396.
- [14] R.M. Adibhatla, J.F. Hatcher, A. Gus, Neur. Res. 37 (2012) 671.
- [15] J. Xuhang, Z. Weigueng, Z. Yum, W. Suclein, Molecules 549 (2002) 553.
- [16] B.S. Furniss, A.J. Hannaford, P.W.G. Smith, A.R. Tatchell, Vogel's Text Book of Practical Organic Chemistry, fifth ed., Pearson Education, London (, 1989.
- [17] Oxford Diffraction CrysAlis PRO, Oxford Diffraction Ltd, Yarnton, England, 2010.
- [18] G.M. Sheldrick, Acta Cryst. A64 (2008) 112.
- [19] A.L. Spek, Acta Cryst. D65 (2009) 148.
- [20] M. Nardelli, J. Appl. Cryst. 28 (1995) 659.
- [21] R.L. Martin, A. Whitley, J. Chem. Soc. (1958) 1394.
- [22] A.B.P. Lever, J. Inorg. Nucl. Chem. 27 (1965) 149.
- [23] D. Coucouvanis, J.P. Fackler, Inorg. Chem. 6 (1967) 2047.
- [24] Z. Travnicek, J. Walla, L.K. Vilk, Z. Sindelar, M. Biler, Trans. Met. Chem. 24 (1999) 6.
- [25] A.B.P. Lever, Inorg. Elect. Spectro. 33 (1984) 507–536.
- [26] D.C. Bradelly, M.H. Gitlitz, J. Chem. Soc. A (1969) 1152-1156.
- [27] N. Dharamraj, P. Viswanathamurthi, K. Natarajan, Trans. Met. Chem. 26 (2001) 105.
- [28] L. Mishra, V.K. Singh, Ind. J. Chem. 32A (1993) 446.
- [29] S. Kapoor, R. Sachar, K. Singh, V.K. Gupta, Rajnikant, J. Chem. Crystallogr. 42 (2012) 222–226.
- [30] S. Kapoor, R. Kour, R. Sachar, R. Kant, V.K. Gupta, K. Kapoor, Acta Cryst. E68 (2012) m58.
- [31] K. Singh, S. Kapoor, R. Sachar, V.K. Gupta, Rajnikant, X-ray Str. Anal. Online 28 (2012) 43–44.
- [32] K. Singh, I. Kour, G. Kour, R. Sachar, V.K. Gupta, Rajnikant, X-ray Str. Anal. Online 29 (2013) 15–16.
- [33] X.H. Jiang, W.G. Zhang, Y. Zhong, S.L. Wang, Molecules 7 (2002) 549-553.
- [34] N. Alam, M.A. Ehsan, M. Zeller, M. Mazhar, Z. Arifin, Acta Cryst. E67 (2011) m1064.