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Data Article

2-(1,3-Oxazolin-2-yl)pyridine and 2,6-bis(1,3-oxazolin-2-yl) pyridine



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ABSTRACT

The data presented in this article are related to research articles “Titanium and vanadium catalysts with oxazoline ligands for ethylene-norbornene (co)polymerization (Ochędzan-Siodłak et al., 2018). For the title compounds, 2-(1,3-oxazolin-2-yl)pyridine (Py-ox) and 2,6-bis(1,3-oxazolin-2-yl)pyridine (Py-box), the single-crystal X-ray diffraction measurement together with NMR, GC, MS, DSC analysis, like also the method of crystallization are presented.

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Specifications table

Subject area	Chemistry
More specific subject area	Organic Chemistry, Ligands for Catalysts
Type of data	Figures, tables, text file. X-ray (table, figures), GC-MS (Figures), 13C NMR (figures), DSC (figures), synthesis (text)

DOI of original article: <https://doi.org/10.1016/j.eurpolymj.2018.07.019>

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How data was acquired	X-ray (Xcalibur diffractometer), NMR (Bruker Ultrashield spectrometer 400 MHz, solvent DMSO-d6), GC-MS (Hewlett Packard HP7890 A GC system) DSC (2010 TA calorimeter)
Data format	X-ray (analyzed), GC-MS (raw), NMR (raw), DSC (raw)
Experimental factors	Crystallization at room temperature. Py-ox - highly anhydrous toluene/ hexane mixture, Py-box - DMSO-d6 in NMR tube.
Experimental features	Highly anhydrous condition for crystals are required.
Data source location	City: Opole, Country: Poland, Latitude: N 50°40'23.981", Longitude: E 17°55'53.173', (Lat,Long: 50.673328, 17.931436999999996),
Data accessibility	The Cambridge Crystallographic Data Centre no. CCDC 1815355 and CCDC 1580983 (http://www.ccdc.cam.ac.uk/conts/retrieving.html , email:deposit@ccdc.cam.ac.uk.).

Value of the data

- X-Ray structural information for Py-ox and Py-box compounds not coordinated by metal atom is presented.
- Conformation and association pattern in the crystal state is shown.
- Crystallization methods are shown.
- Purification for Py-ox is improved.

1. Data

The presented compounds, 2-(1,3-oxazolin-2-yl)pyridine (Py-ox) and 2,6-bis(1,3-oxazolin-2-yl)pyridine (Py-box), are commonly applied as ligands for complexes with transition metals: cobalt [2], rhenium [3], platinum and palladium [4,5] for Py-ox, as well as copper [6,7], ruthenium [8–11], rhodium [12], manganese [13], silver [14], nickel [15], cobalt [16], terbium [17], and iron [18], in the case of Py-box. Some of them reveal catalytic properties. In our work, the Py-ox and Py-box compounds were applied as ligands for titanium and vanadium complexes, which turned out to be active in polymerization of ethylene and copolymerization of ethylene with norbornene [1]. The X-Ray information for Py-ox and Py-box compounds can be important for comparative studies, to show differences between these compounds not coordinated by metal atom and applied as ligands. It can help to understand dependence between the structure and activity of the designed complexes. The presented crystallization methods are worth to notice. The improved method of purification enable to obtain the studied compound of high quality.

2. Experimental design, materials and methods

2.1. Synthesis

2.1.1. 2-(1,3-oxazolin-2-yl)pyridine (Py-ox)

The synthesis was performed mainly according to Stokes et al. [19]. The crude product was subjected to flash chromatography using the MeOH: AcOEt (1:4) mixture as eluent. Yield 60%. Elemental analysis C₈H₈N₂O results: calculated C 64.85%, H 5.44%, N 18.91%, experimental C 64.92%, H 5.45%, N 19.09%. ¹H NMR (400 MHz, DMSO-d6) δ 8.65 (1H, J = 4.5 Hz, d), 7.99 (1H, J = 8.0 Hz, d), 7.93 (1H, J = 7.8 Hz, td), 7.54 (1H, m), 4.45 (2H, J = 9.6 Hz, t), 4.00 (2H, J = 9.6 Hz, t). ¹³C NMR (400 MHz, DMSO-d6) δ 162.98, 149.53, 146.52, 137.09, 125.90, 123.80, 67.66, 54.61. GC-MS M⁺ 148 m/e. Melting temperature 57.0 (54.6–60.0) °C.

2.1.2. 2,6-bis(1,3-oxazolin-2-yl)pyridine (Py-box)

The synthesis was performed mainly according to Zhu et al. [20]. Yield 76%. Elemental analysis $C_{11}H_{11}N_3O_2$ results: calculated C 64.82%, H 5.10%, N 19.34%, experimental: C 64.88%, H 5.12%, N 19.39%. 1H NMR (400 MHz, DMSO-*d*6) δ 8.11 (2H, $J_1 = 1.2$ Hz, $J_2 = 7.2$ Hz, t), 8.02(1H, $J_1 = 6.4$ Hz, $J_2 = 2.4$ Hz, q), 4.45 (4H, $J = 9.6$ Hz, t), 4.01 (4H, $J = 9.6$ Hz, t). ^{13}C NMR (400 MHz, DMSO-*d*6) δ 163.10, 147.01, 138.46, 126.00, 68.28, 55.13. GC-MS M^+ 217 m/e. Melting temperature 160.6 (159.4–163.0) °C.

2.2. Crystallization

2.2.1. 2-(1,3-oxazolin-2-yl)pyridine (Py-ox)

The crystals were obtained at room temperature from highly anhydrous toluene/hexane mixture. The solvents were freshly distilled over sodium. The highly anhydrous conditions are crucial. All operations were performed in a glove-box filled with argon. Py-ox (20 mg) was placed in a 5 ml snap cap vial with plastic cap and dissolved in toluene (1 ml). Then, hexane (1 ml) was added and the solution was left to stand at room temperature for a week.

2.2.2. 2,6-bis(1,3-oxazolin-2-yl)pyridine (Py-box)

The crystals of appropriate quality were obtained at room temperature from DMSO-*d*6 solution by long standing time in NMR tube. All operations were performed in a glove-box filled with argon. DMSO-*d*6 solvent from sealed glass ampoules was applied. Py-box (15 mg) and DMSO-*d*6 (0.6 ml) was placed in NMR tube and the cap was sealed by a parafilm. The solution was left to stand at room temperature for a month.

2.3. X-ray

The single-crystal X-ray diffraction experiments were performed at 293.0(1)K on the Xcalibur diffractometer, equipped with a CCD area detector and a graphite monochromator for the MoK α

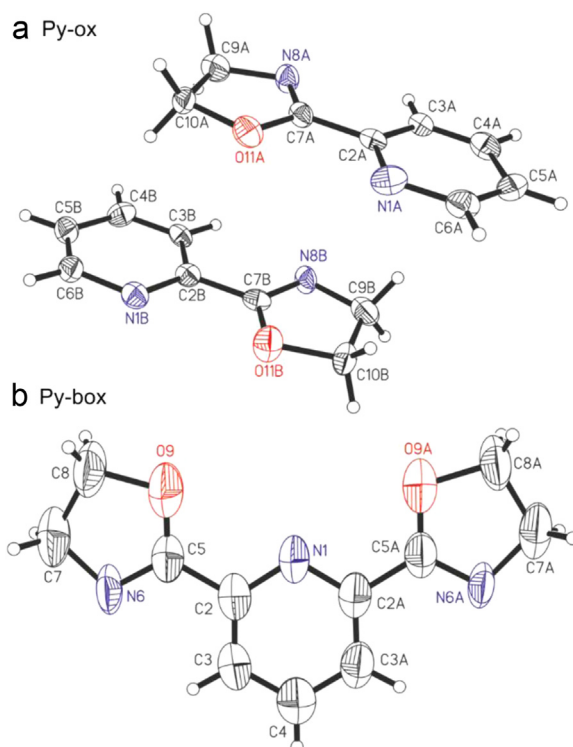


Fig. 1. Molecular conformation of Py-ox (a) and Py-box (b) with atom labeling and the displacement ellipsoids at 50% probability level.

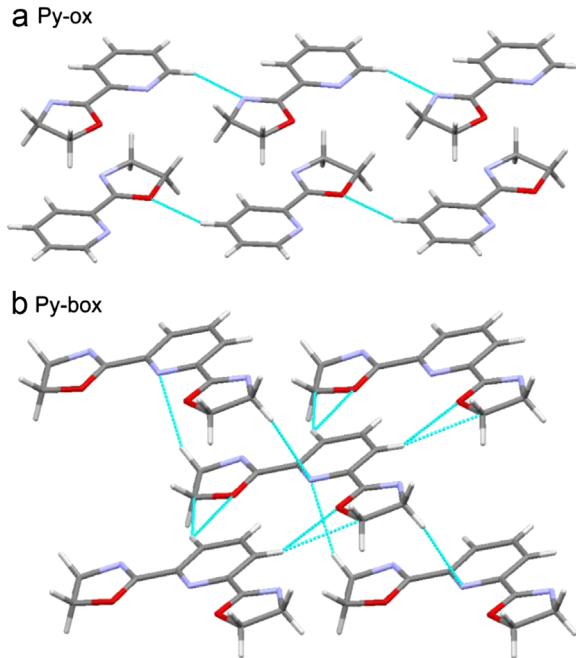


Fig. 2. Association of molecule in the crystal structure. Hydrogen contacts are marked by dashed lines. The numbers of atoms and distances are omitted for clarity. All geometric parameters are in Table 2.

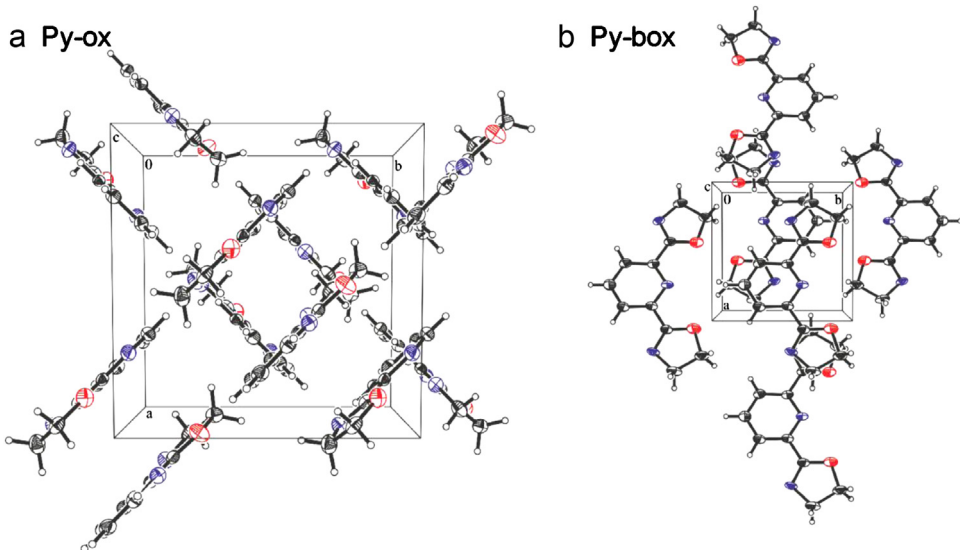


Fig. 3. The crystal packing scheme of the title compounds. A view along the c axis of the crystals packing.

radiation. The reciprocal space was explored by ω scans with detector positions at 60 mm distance from the crystal. The diffraction data processing of studied compounds (Lorentz and polarization corrections were applied) were performed using the CrysAlis CCD [21,22]. Both structures Py-ox and Py-box were solved in the C2 and P2/n space group respectively, by direct methods and refined by a

Table 1

X-ray experimental details for 2-(1,3-oxazolin-2-yl)pyridine (Py-ox) and 2,6-bis(1,3-oxazolin-2-yl) pyridine (Py-box).

	Py-ox	Py-box
Chemical formula	C ₈ H ₈ N ₂ O	C ₁₁ H ₁₁ N ₃ O ₂
<i>M_r</i>	148.16	217.23
Crystal system, space group	Monoclinic, <i>C2</i>	Monoclinic, <i>P2₁/n</i>
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.2571 (7), 10.0159 (6), 14.4647 (9)	6.4904 (8), 6.5835 (11), 11.9080 (19)
β (°)	97.497 (6)	94.215 (13)
<i>V</i> (Å ³)	1473.31 (16)	507.45 (13)
<i>Z</i>	8	2
Measurement temperature	293.0(1)	293.0(1)
μ (mm ⁻¹)	0.09	0.10
Crystal size (mm)	0.4 × 0.3 × 0.2	0.5 × 0.4 × 0.3
Crystal colour	Colourless	
Crystal description	Plate	
Data collection		
Radiation wavelength	0.71073	
Radiation type	MoKα	
Source	fine-focus sealed tube	
Measurement device type	Xcalibur	
Detector area resolution	1024 × 1024 with blocks 2 × 2	
Absorption correction	–	
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	5,034, 2786, 1587	3,172, 993, 459
<i>R</i> _{int}	0.018	0.048
(sin θ/λ) _{max} (Å ⁻¹)	0.617	0.616
Refinement		
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.030, 0.077, 0.86	0.057, 0.173, 0.87
No. of reflections	2786	993
No. of parameters	200	75
No. of restraints	1	0
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.11, –0.09	0.22, –0.18

Table 2

Selected geometric parameters (Å, °) for Py-ox and Py-box molecules.

Structure 2 (Py-ox)			
N1A–C2A	1.386 (7)	C5B–H5B	0.9300
N1A–C6A	1.394 (6)	C7A–N8A	1.292 (7)
C2A–C3A	1.367 (8)	C7A–O11A	1.314 (6)
C2A–H2A	0.9300	C6B–C7B	1.478 (7)
N1B–C6B	1.360 (6)	N8A–C9A	1.423 (7)
N1B–C2B	1.393 (7)	C7B–N8B	1.289 (6)
C3A–C4A	1.400 (8)	C7B–O11B	1.292 (7)
C3A–H3A	0.9300	C9A–C10A	1.518 (8)
C2B–C3B	1.344 (9)	C9A–H9AA	0.9700
C2B–H2B	0.9300	C9A–H9AB	0.9700
C4A–C5A	1.307 (8)	N8B–C9B	1.427 (7)
C4A–H4A	0.9300	C10A–O11A	1.470 (7)
C3B–C4B	1.361 (9)	C10A–H10A	0.9700
C3B–H3B	0.9300	C10A–H10B	0.9700
C5A–C6A	1.335 (6)	C9B–C10B	1.513 (8)
C5A–H5A	0.9300	C9B–H9BA	0.9700
C4B–C5B	1.345 (7)	C9B–H9BB	0.9700
C4B–H4B	0.9300	O11B–C10B	1.488 (6)
C6A–C7A	1.464 (7)	C10B–H10C	0.9700
C5B–C6B	1.342 (6)	C10B–H10D	0.9700
C2A–N1A–C6A	116.2 (5)	C5B–C6B–C7B	119.1 (5)

Table 2 (continued)

Structure 2 (Py-ox)			
C3A-C2A-N1A	122.5 (6)	N1B-C6B-C7B	117.4 (5)
C3A-C2A-H2A	118.8	C7A-N8A-C9A	106.3 (5)
N1A-C2A-H2A	118.8	N8B-C7B-O11B	119.6 (6)
C6B-N1B-C2B	115.8 (5)	N8B-C7B-C6B	120.4 (6)
C2A-C3A-C4A	116.1 (6)	O11B-C7B-C6B	120.0 (5)
C2A-C3A-H3A	122.0	N8A-C9A-C10A	106.9 (6)
C4A-C3A-H3A	122.0	N8A-C9A-H9AA	110.3
C3B-C2B-N1B	121.2 (6)	C10A-C9A-H9AA	110.3
C3B-C2B-H2B	119.4	N8A-C9A-H9AB	110.3
N1B-C2B-H2B	119.4	C10A-C9A-H9AB	110.3
C5A-C4A-C3A	123.3 (6)	H9AA-C9A-H9AB	108.6
C5A-C4A-H4A	118.3	C7B-N8B-C9B	105.7 (5)
C3A-C4A-H4A	118.3	O11A-C10A-C9A	101.9 (4)
C2B-C3B-C4B	120.0 (6)	O11A-C10A-H10A	111.4
C2B-C3B-H3B	120.0	C9A-C10A-H10A	111.4
C4B-C3B-H3B	120.0	O11A-C10A-H10B	111.4
C4A-C5A-C6A	119.6 (6)	C9A-C10A-H10B	111.4
C4A-C5A-H5A	120.2	H10A-C10A-H10B	109.3
C6A-C5A-H5A	120.2	N8B-C9B-C10B	106.8 (4)
C5B-C4B-C3B	120.2 (6)	N8B-C9B-H9BA	110.4
C5B-C4B-H4B	119.9	C10B-C9B-H9BA	110.4
C3B-C4B-H4B	119.9	N8B-C9B-H9BB	110.4
C5A-C6A-N1A	122.4 (5)	C10B-C9B-H9BB	110.4
C5A-C6A-C7A	118.1 (5)	H9BA-C9B-H9BB	108.6
N1A-C6A-C7A	119.6 (5)	C7A-O11A-C10A	106.8 (5)
C6B-C5B-C4B	119.1 (5)	C7B-O11B-C10B	106.1 (5)
C6B-C5B-H5B	120.5	O11B-C10B-C9B	101.7 (5)
C4B-C5B-H5B	120.5	O11B-C10B-H10C	111.4
N8A-C7A-O11A	118.1 (5)	C9B-C10B-H10C	111.4
N8A-C7A-C6A	122.8 (5)	O11B-C10B-H10D	111.4
O11A-C7A-C6A	119.1 (6)	C9B-C10B-H10D	111.4
C5B-C6B-N1B	123.6 (5)	H10C-C10B-H10D	109.3
Symmetry code(s): (i) $-x+1/2, y, -z+1/2$.			
Structure 1 (Py-Box)			
N1-C2 ¹	1.355 (3)	C5-O9	1.316 (4)
N1-C2	1.355 (3)	N6-C7	1.448 (4)
C2-C3	1.381 (4)	C7-C8	1.502 (4)
C2-C5	1.468 (4)	C7-H7A	0.9700
C3-C4	1.380 (4)	C7-H7B	0.9700
C3-H3	0.9300	C8-O9	1.471 (3)
C4-C3 ¹	1.380 (4)	C8-H8A	0.9700
C4-H4	0.9300	C8-H8B	0.9700
C5-N6	1.293 (3)		
C2 ¹ -N1-C2	116.0 (4)	N6-C7-C8	105.1 (2)
N1-C2-C3	123.4 (3)	N6-C7-H7A	110.7
N1-C2-C5	116.5 (3)	C8-C7-H7A	110.7
C3-C2-C5	120.0 (2)	N6-C7-H7B	110.7
C4-C3-C2	119.5 (3)	C8-C7-H7B	110.7
C4-C3-H3	120.2	H7A-C7-H7B	108.8
C2-C3-H3	120.2	O9-C8-C7	104.0 (3)
C3 ¹ -C4-C3	118.1 (4)	O9-C8-H8A	110.9
C3 ¹ -C4-H4	121.0	C7-C8-H8A	110.9
C3-C4-H4	121.0	O9-C8-H8B	110.9
N6-C5-O9	118.1 (2)	C7-C8-H8B	110.9
N6-C5-C2	121.0 (3)	H8A-C8-H8B	109.0
O9-C5-C2	120.9 (2)	C5-O9-C8	105.8 (2)
C5-N6-C7	106.9 (2)		

full-matrix least-squares method using SHELXL14 program [23,24]. The H atoms were found based on geometrical parameters. In both structures H atoms were refined using a riding model. The structure drawings were prepared using SHELXTL and Mercury programs [25] (Figs. 1–3 and Tables 1,2).

2.3.1. 2-(1,3-oxazolin-2-yl)pyridine (Py-ox)

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) bd203_c

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No syntax errors found. CIF dictionary Interpreting this report

Datablock: bd203_c

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Hall group	C 2y	C 2y	
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F000'	624.25		
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Nref	2899 [1538]	2786	
Tmin, Tmax	0.997, 0.998		
Tmin'	0.996		
Correction method= Not given			
Data completeness=	1.81/0.96	Theta (max)= 25.997	
R (reflections)=	0.0304 (1587)	wR2 (reflections)= 0.0771 (2786)	
S =	0.863	Npar= 200	

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
 Click on the hyperlinks for more details of the test.

Alert level B

PLAT111_ALERT_2_B	ADDSYM Detects New (Pseudo) Centre of Symmetry .	100	%Fit
PLAT112_ALERT_2_B	ADDSYM Detects New (Pseudo) Symm. Elem c	100	%Fit
PLAT113_ALERT_2_B	ADDSYM Suggests Possible Pseudo/New Space Group	C2/c	Check
PLAT230_ALERT_2_B	Hirshfeld Test Diff for N8B --C7B	8.3	s.u.
PLAT230_ALERT_2_B	Hirshfeld Test Diff for N8B --C9B	8.2	s.u.

Alert level C

STRVA01_ALERT_4_C	Flack parameter is too small		
	From the CIF: <code>_refine_ls_abs_structure_Flack</code>	-1.100	
	From the CIF: <code>_refine_ls_abs_structure_Flack_su</code>	1.000	
PLAT230_ALERT_2_C	Hirshfeld Test Diff for O11A --C10A	5.2	s.u.
PLAT230_ALERT_2_C	Hirshfeld Test Diff for N8A --C7A	5.2	s.u.
PLAT230_ALERT_2_C	Hirshfeld Test Diff for C3A --C4A	7.0	s.u.
PLAT230_ALERT_2_C	Hirshfeld Test Diff for O11B --C10B	5.8	s.u.
PLAT234_ALERT_4_C	Large Hirshfeld Difference N8A --C9A	0.17	Ang.
PLAT241_ALERT_2_C	High 'MainMol' Ueq as Compared to Neighbors of O11A	Check	
PLAT241_ALERT_2_C	High 'MainMol' Ueq as Compared to Neighbors of N1A	Check	
PLAT241_ALERT_2_C	High 'MainMol' Ueq as Compared to Neighbors of C9A	Check	
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PLAT242_ALERT_2_C	Low 'MainMol' Ueq as Compared to Neighbors of C10A	Check	
PLAT340_ALERT_3_C	Low Bond Precision on C-C Bonds	0.00842	Ang.
PLAT411_ALERT_2_C	Short Inter H...H Contact H3A ..H3A	2.12	Ang.
	1-x,y,1-z =	2_656	Check
PLAT911_ALERT_3_C	Missing FCF Refl Between Thmin & STh/L=	0.600	2 Report
PLAT978_ALERT_2_C	Number C-C Bonds with Positive Residual Density.	0	Info

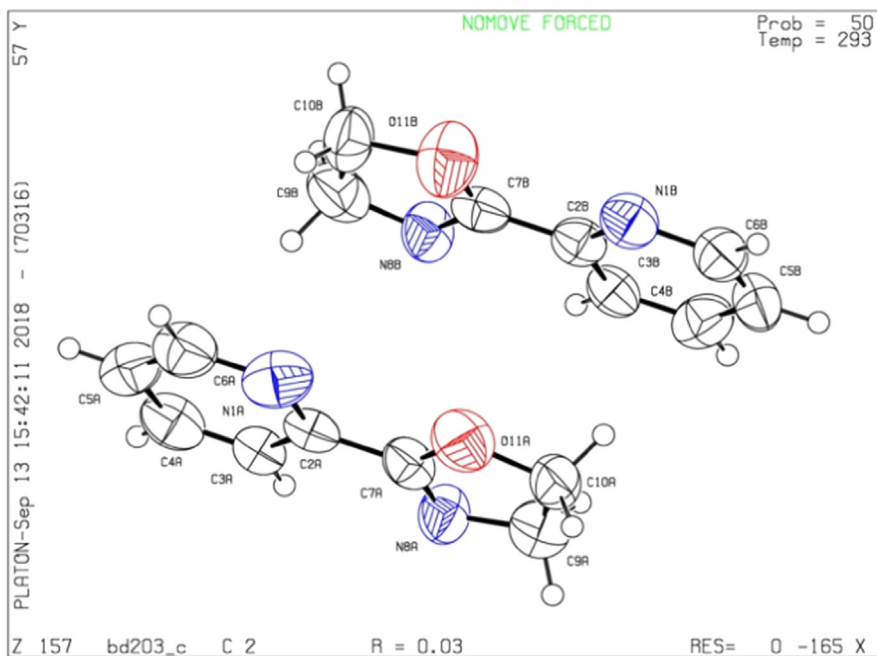
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PLAT200_ALERT_1_G	Reported <code>_diffn_ambient_temperature</code> (K)	293	Check
PLAT398_ALERT_2_G	Deviating C-O-C Angle From 120 for O11A	106.8	Degree
PLAT398_ALERT_2_G	Deviating C-O-C Angle From 120 for O11B	106.0	Degree
PLAT720_ALERT_4_G	Number of Unusual/Non-Standard Labels	4	Note
PLAT910_ALERT_3_G	Missing # of FCF Reflection(s) Below Theta(Min).	3	Note

- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
 5 **ALERT level B** = A potentially serious problem, consider carefully
 15 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
 7 **ALERT level G** = General information/check it is not something unexpected

- 2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 18 ALERT type 2 Indicator that the structure model may be wrong or deficient
 3 ALERT type 3 Indicator that the structure quality may be low
 4 ALERT type 4 Improvement, methodology, query or suggestion
 0 ALERT type 5 Informative message, check

Datablock bd203_c - ellipsoid plot



2.3.2. 2,6-bis(1,3-oxazolin-2-yl)pyridine (Py-box)

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) BD162_a

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No syntax errors found. CIF dictionary Interpreting this report

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	Calculated	Reported	
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Space group	P 2/n	P 2/n	
Hall group	-P 2yac	-P 2yac	
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Sum formula	C11 H11 N3 O2	C11 H11 N3 O2	
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Dx, g cm ⁻³	1.422	1.422	
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F000'	228.10		
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Nref	992	993	
Tmin, Tmax	0.995, 0.997		
Tmin'	0.995		

Correction method= Not given

Data completeness= 1.001 Theta(max)= 25.982

R(reflections)= 0.0572 (459) wR2(reflections)= 0.1732 (993)

S = 0.870 Npar= 75

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
 Click on the hyperlinks for more details of the test.

Alert level C

PLAT026_ALERT_3_C	Ratio Observed / Unique Reflections (too) Low ..	46 %
PLAT241_ALERT_2_C	High 'MainMol' Ueq as Compared to Neighbors of	09 Check
PLAT250_ALERT_2_C	Large U3/U1 Ratio for Average U(i,j) Tensor	2.7 Note
PLAT340_ALERT_3_C	Low Bond Precision on C-C Bonds	0.0045 Ang.
PLAT906_ALERT_3_C	Large K value in the Analysis of Variance	13.338 Check
PLAT978_ALERT_2_C	Number C-C Bonds with Positive Residual Density.	0 Info

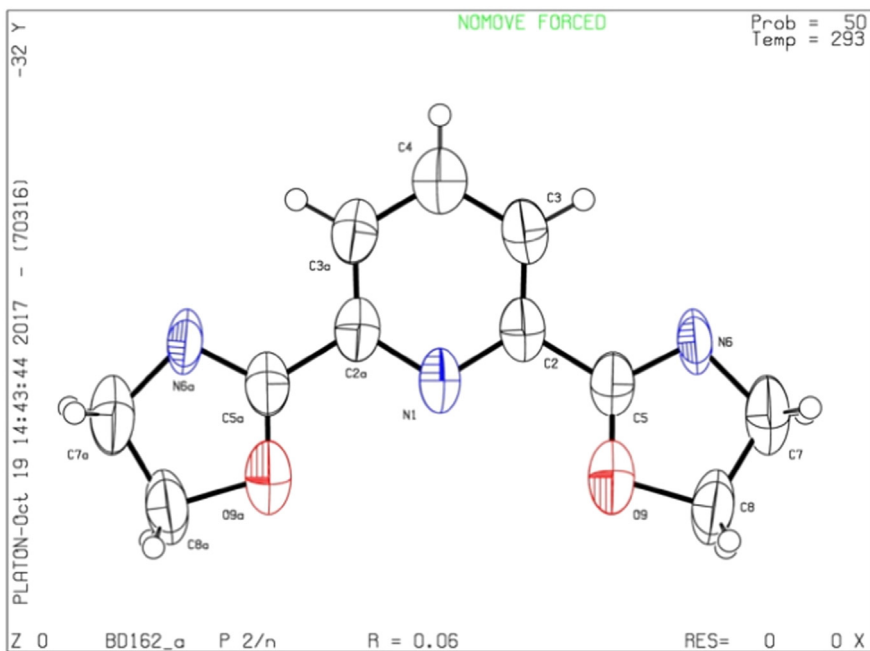
Alert level G

PLAT072_ALERT_2_G	SHELXL First Parameter in WGHT Unusually Large	0.10 Report
PLAT199_ALERT_1_G	Reported _cell_measurement_temperature	293 Check
PLAT200_ALERT_1_G	Reported _diffrn_ambient_temperature	293 Check
PLAT398_ALERT_2_G	Deviating C-O-C Angle from 120 Deg for O9	105.9 Degree
PLAT953_ALERT_1_G	Reported (CIF) and Actual (FCF) Hmax Differ by .	1 Units

- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
 0 **ALERT level B** = A potentially serious problem, consider carefully
 6 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
 5 **ALERT level G** = General information/check it is not something unexpected
- 3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 5 ALERT type 2 Indicator that the structure model may be wrong or deficient
 3 ALERT type 3 Indicator that the structure quality may be low
 0 ALERT type 4 Improvement, methodology, query or suggestion
 0 ALERT type 5 Informative message, check

PLATON version of 13/08/2017; check.def file version of 27/07/2017

Datablock BD162_a - ellipsoid plot



2.4. NMR

Bruker Ultrashield spectrometer 400 MHz, solvent DMSO-d₆, TMS standard. Concentration: 15 mg in 0.6 ml (Figs. 4–7).

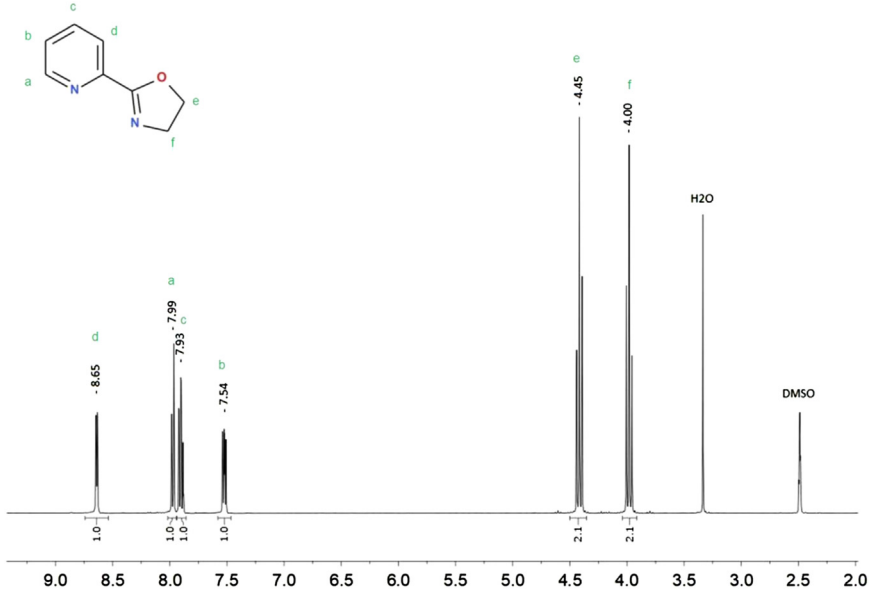


Fig. 4. ¹H NMR spectrum for 2-(1,3-oxazolin-2-yl)pyridine (Py-ox) in DMSO-d₆.

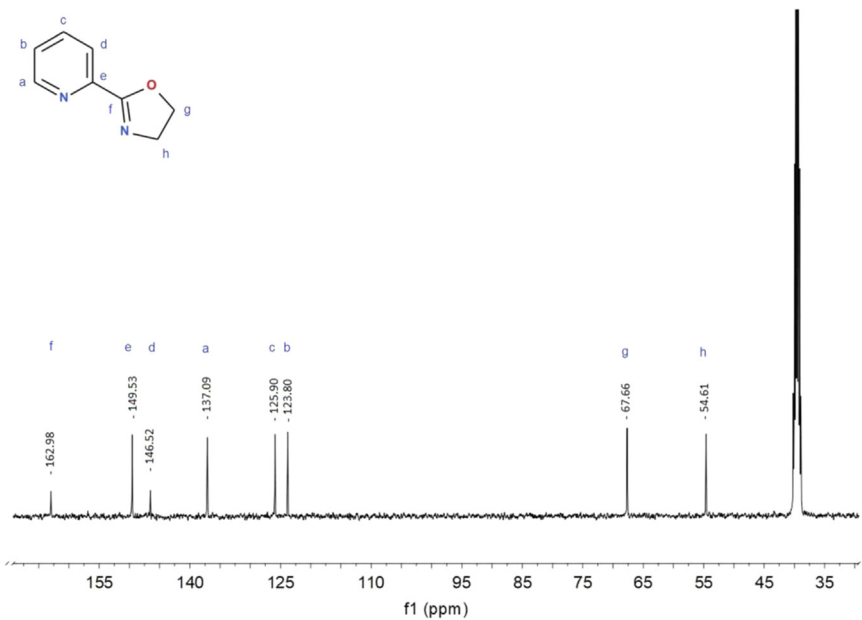


Fig. 5. ¹³C NMR spectrum for 2-(1,3-oxazolin-2-yl)pyridine (Py-ox) in DMSO-d₆.

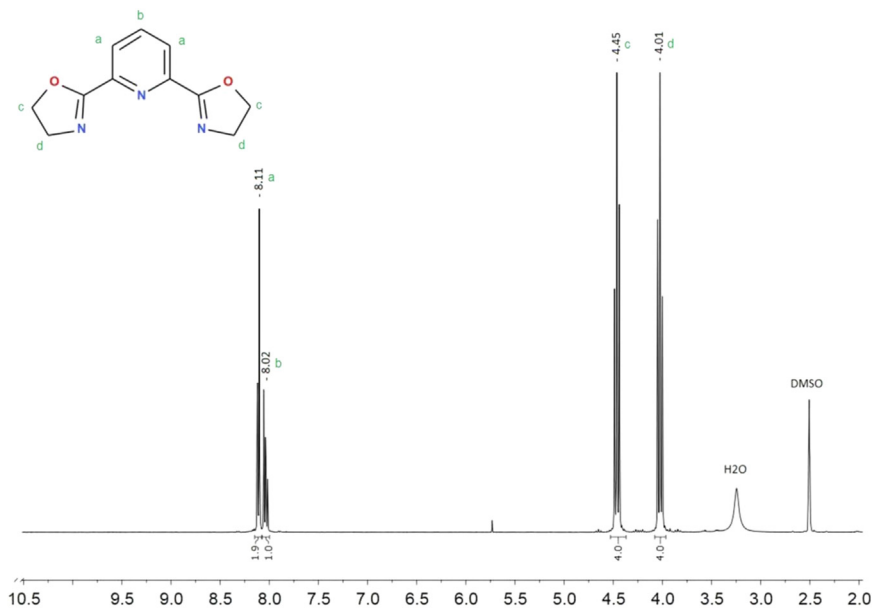


Fig. 6. ¹H NMR spectrum for 2,6-bis(1,3-oxazolin-2-yl)pyridine (Py-box) in DMSO-d₆.

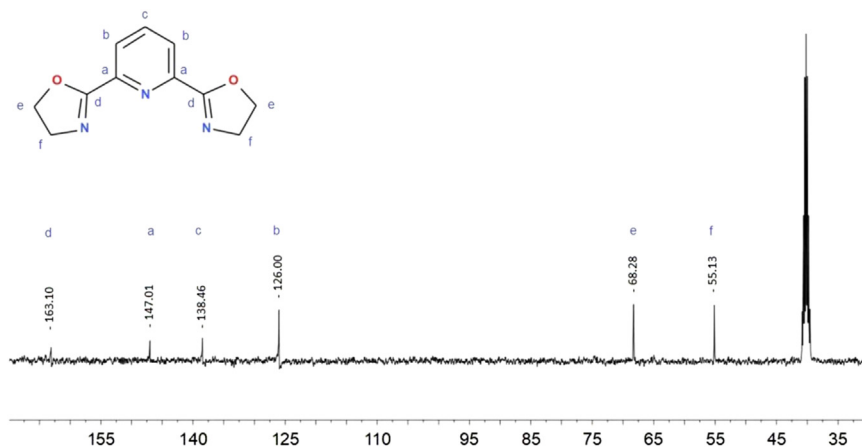


Fig. 7. ¹³C NMR spectrum for 2,6-bis(1,3-oxazolin-2-yl)pyridine (Py-box) in DMSO-d₆.

2.5. GC–MS

Hewlett Packard HP7890 A GC system, equipped with 7000 GC/MS triple-quadrupol and HP-5 capilar 300 m × 0.32 mm column with 0.25 μm dimethylpolysiloxane stationary phase, dopped by 5% of phenylpolysiloxane (Figs. 8–11).

2.6. DSC

The melting temperatures were measured by differential scanning calorimetry DSC 2010 TA instrument calorimeter equipped with an automated sampler. The data were collected with the heat/cool/heat cycle at a heating rate of 10 °C/min under a nitrogen atmosphere (Figs. 12 and 13).

Sample Name		Position	1	Instrument Name	7000A	User Name	DATASYSTEM01\Admin
Inj Vol	0	InjPosition		SampleType		IRM Calibration Status	Not Applicable
Data Filename	PyOzn_ACN_p1_17.D	ACQ Method	MULTI_ALS1.M	Comment	80-1/20/300 inj.320 sd3 HTS	Acquired Time	5/16/2017 3:12:34 PM

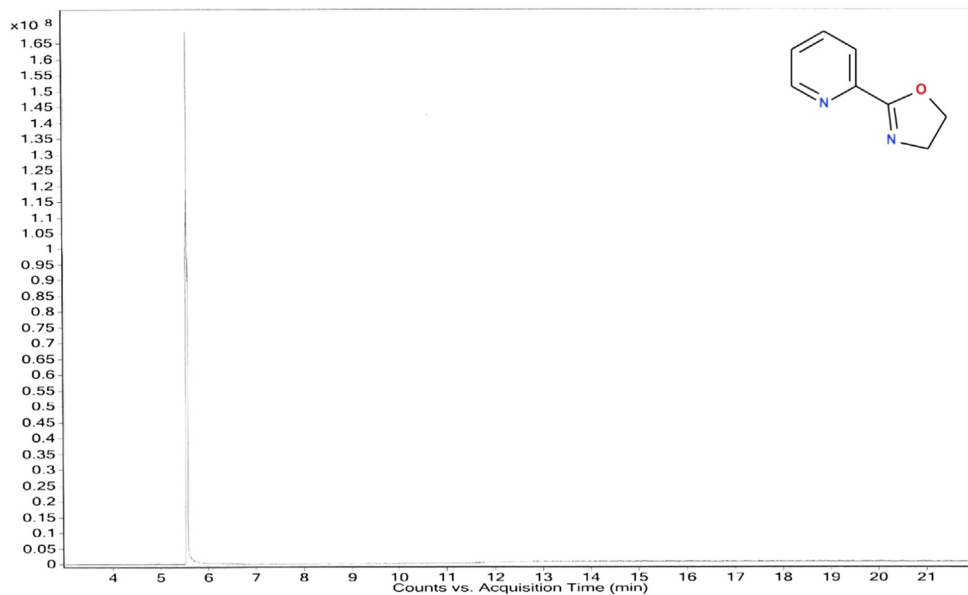


Fig. 8. GC analysis of 2-(1,3-oxazolin-2-yl)pyridine (Py-ox).

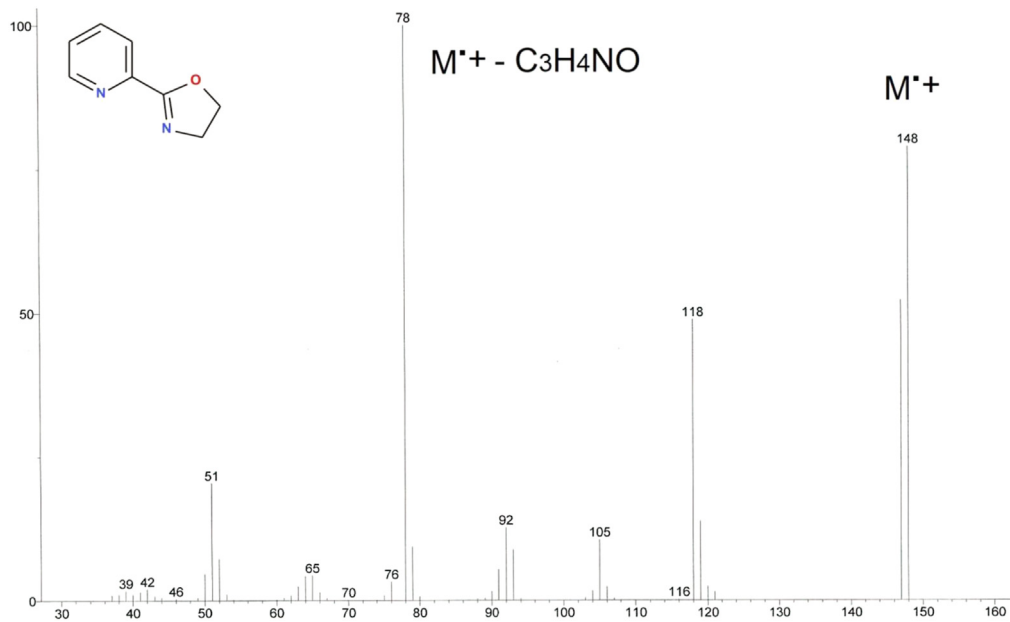


Fig. 9. MS analysis of 2-(1,3-oxazolin-2-yl)pyridine (Py-ox).

Sample Name	Position	1	Instrument Name	7000A	User Name	DATASYSTEM01\Admin	
Inj Vol	0	InjPosition	SampleType		IRM Calibration Status	Not Applicable	
Data Filename	PYBox_ACN_p1_17.D	ACQ Method	MULTI_ALS1.M	Comment	80-1/20/300 inj.320 sd3 HTS	Acquired Time	5/16/2017 2:47:40 PM

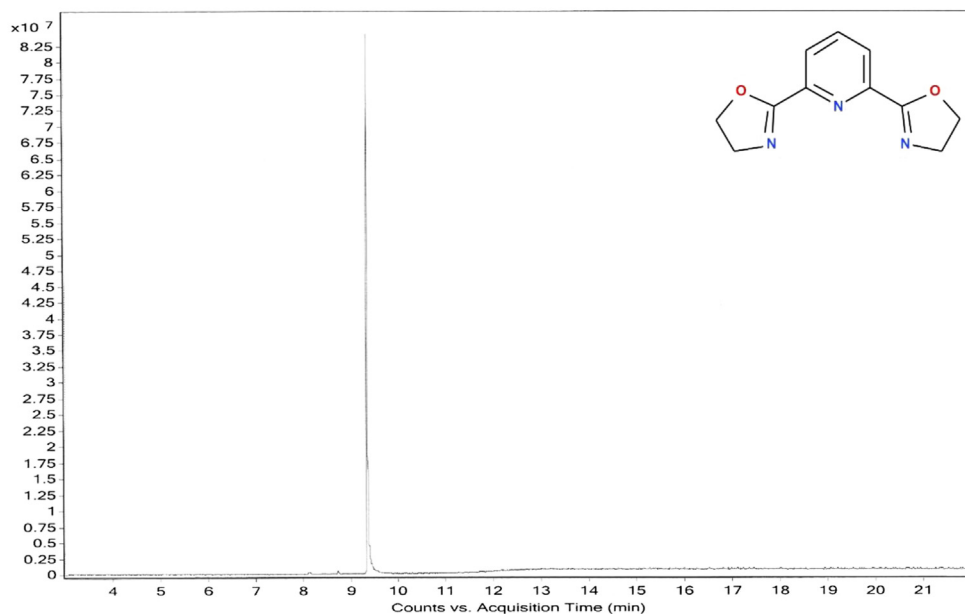


Fig. 10. GC analysis of 2,6-bis(1,3-oxazolin-2-yl)pyridine (Py-box).

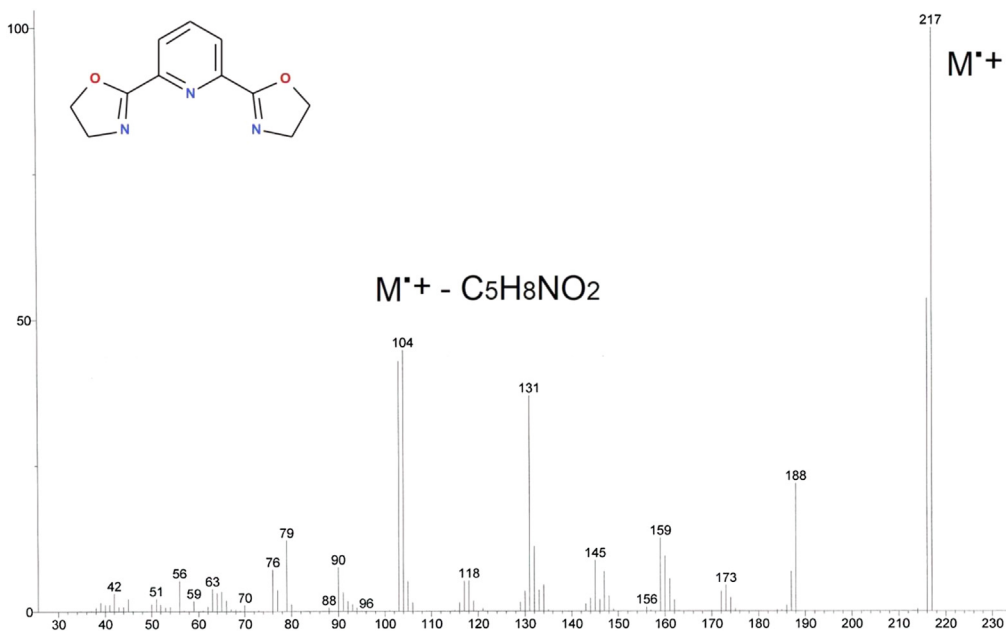


Fig. 11. MS analysis of 2,6-bis(1,3-oxazolin-2-yl)pyridine (Py-box).

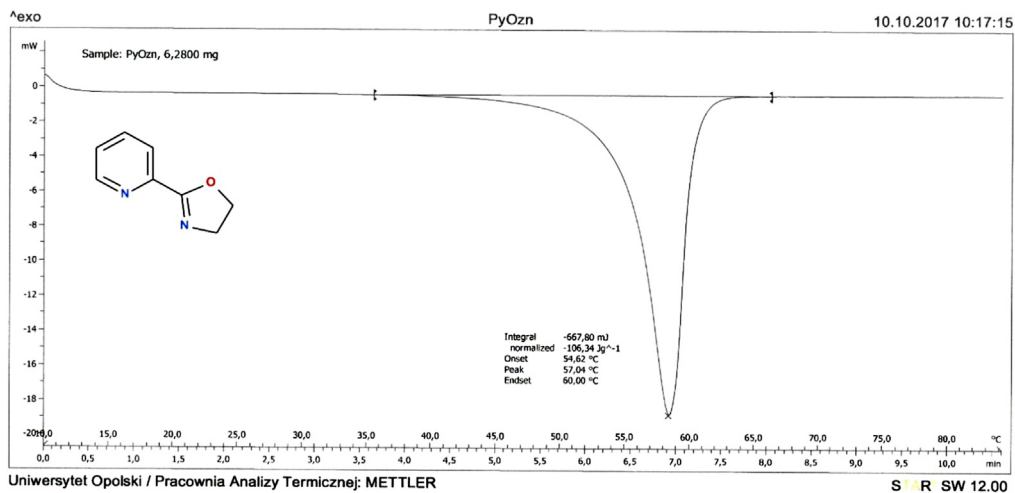


Fig. 12. DSC analysis of 2-(1,3-oxazolin-2-yl)pyridine (Py-ox).

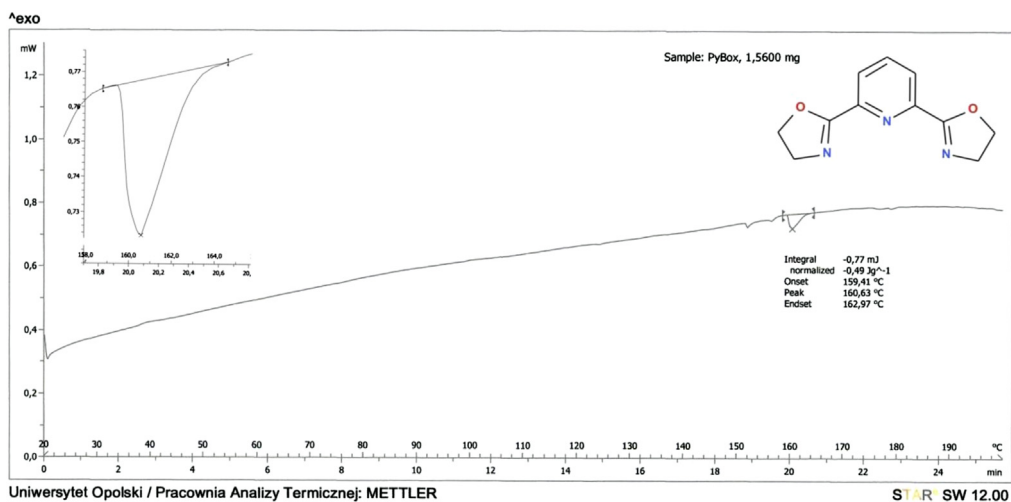


Fig. 13. DSC analysis of 2,6-bis(1,3-oxazolin-2-yl)pyridine (Py-box).

Transparency document. Supporting information

Transparency data associated with this article can be found in the online version at <https://doi.org/10.1016/j.dib.2018.09.129>.

References

- [1] W. Ochędzan-Siodlak, A. Bihun-Kisiel, D. Siodlak, A. Poliwoda, B. Dziuk, Titanium and vanadium catalysts with oxazoline ligands for ethylenenorbornene (co)polymerization, *Eur. Polym. J.* 106 (2018) 148–155.
- [2] J. Guo, H. Liu, J. Bi, C. Zhang, H. Zhang, C. Bai, Y. Hu, X. Zhang, Pyridine-oxazoline and quinoline-oxazoline ligated cobalt complexes: synthesis, characterization, and 1,3-butadiene polymerization behaviors, *Inorganica Chim. Acta* 435 (2015) 305–312.

- [3] J.K. Nganga, C.R. Samanam, J.M. Tanski, C. Pacheco, C. Saucedo, V.S. Batista, K.A. Grice, M.Z. Ertem, A.M. Angeles-Boza, Electrochemical reduction of CO₂ catalyzed by Re(pyridineoxazoline)(CO)₃Cl, *Complexes Inorg. Chem.* 56 (2017) 3214–3226.
- [4] N. Paschke, A. Rödigs, H. Poppenborg, J.E.A. Wolff, B. Krebs, Reaction of the diaqua(2-(pyridin-2-yl)-2-oxazoline)platinum(II) and -palladium(II) dications with the model nucleobases 1-methylthymine and 1-methyluracil: syntheses, spectroscopic properties and X-ray crystal structures, *Inorganica Chim. Acta* 264 (1997) 239–248.
- [5] B.S. Fedorov, N.I. Golovina, G.V. Strukov, V.V. Kedrov, G.N. Boiko, G.V. Shilov, L.S. Barinova, R.F. Trofimova, L.O. Atovmyan, Synthesis and the crystal structures of N-(2-nitroxyethyl)isonicotinamide and its complexes with PdCl₂ and PtCl₂ as potential antitumor medicines, *Russ. Chem. Bull.* 50 (2001) 520–524.
- [6] H.W. Kuai, X.C. Cheng, D.H. Li, T. Hu, X.H. Zhu, Syntheses, characterization and properties of silver, copper and palladium complexes from bis(oxazoline)-containing ligands, *J. Solid State Chem.* 228 (2015) 65–75.
- [7] Y.Y. Zhu, C. Cui, N. Li, B.W. Wang, Z.M. Wang, S. Gao, Constructing a series of azide-bridged Cu^{II} magnetic low-dimensional coordination polymers by using Pybox ligands, *Eur. J. Inorg. Chem.* 17 (2013) 3101–3111.
- [8] D. Doberer, C. Slugovc, R. Schmid, K. Kirchner, K. Mereiter, Coordination chemistry of 2,6-bis(oxazoliny)-pyridine ruthenium complexes, *Monatsh. Chem.* 130 (1999) 717–723.
- [9] D. Cuervo, E. Menendez-Pedregal, J. Diez, M.P. Gama, Mononuclear ruthenium(II) complexes bearing the (S,S)-iPr-pybox ligand, *J. Organomet. Chem.* 696 (2011) 1861–1867.
- [10] Y. Motoyama, O. Kurihara, K. Murata, K. Aoki, H. Nishiyama, Chiral ruthenium–bis(oxazoliny)pyridine complexes of α,β -unsaturated carbonyl compounds: enantioface-selective coordination of olefins, *Organometallics* 19 (2000) 1025–1034.
- [11] H. Nishiyama, Y. Itoh, Y. Sugawara, H. Matsumoto, K. Aoki, K. Itoh, Chiral ruthenium(ii)-bis(2-oxazolin-2-yl)pyridine complexes. Asymmetric catalytic cyclopropanation of olefins and diazoacetates, *Bull. Chem. Soc. Jpn.* 68 (1995) 1247–1262.
- [12] H. Nishiyama, E. Niwa, T. Inoue, Y. Ishima, K. Aoki, Novel metallacycle complexes from bis(oxazoliny)pyridine – rhodium (i) species and diynes, *Organometallics* 21 (2002) 2572–2574.
- [13] N. Liu, L.-H. Jia, Z.-Q. Wu, Y.-Y. Zhu, B.-W. Wang, S. Gao, Wujia Huaxue Xuebao (Chin. J. Inorg. Chem.) 30 (2014) 1660.
- [14] C.H. Dai, F.L. Mao, Z. Naturforsch, Helical chain Ag(I) complexes with a tridentate N-donor ligand: syntheses, structural characterization, and properties, *Z. Naturforsch. B: J. Chem. Sci.* 70 (2015) 851–856.
- [15] Y.H. Zhang, Y. Zahng, Q. Yue, C.G. Gan, Synthesis, characterization and crystal structure of Ni(Pybox)(SCN)₂(CH₃OH), *Huaxue Yanjiu Yu Yingyong*(Chem. Res. Appl.) 23 (2011) 1525 (Chin).
- [16] J. Guo, B. Wang, J. Bi, C. Zhang, H. Zhang, C. Bai, Y. Hu, X. Zhang, Synthesis, characterization and 1,3-butadiene polymerization studies of cobalt dichloride complexes bearing pyridine bisoxazoline ligands, *Polymer* 59 (2015) 124–132.
- [17] A. de Bettencourt-Dias, P.S. Barber, S. Viswanathan, D.T. de Lill, A. Rollett, G. Ling, S. Altun, Para-derivatized pybox ligands as sensitizers in highly luminescent Ln(III) complexes, *Inorg. Chem.* 49 (2010) 8848–8861.
- [18] Y.Y. Zhu, H.Q. Li, Z.Y. Ding, X.J. Lu, L. Zhao, Y.S. Meng, T. Liu, S. Gao, Spin transitions in a series of [Fe(pybox)₂]²⁺ complexes modulated by ligand structures, counter anions, and solvents, *Inorg. Chem. Front.* 3 (2016) 1624–1636.
- [19] B.J. Stokes, S.M. Opra, M.S. Sigman, Palladium-catalyzed allylic cross-coupling reactions of primary and secondary homoallylic electrophiles, *J. Am. Chem. Soc.* 134 (2012) 11408–11411.
- [20] Y.Y. Zhu, C. Cui, N. Li, B.W. Wang, Z.M. Wang, S. Gao, Constructing a series of azide-bridged Cu^{II} magnetic low-dimensional coordination polymers by using pybox ligands, *Eur. J. Inorg. Chem.* 17 (2013) 3101–3111.
- [21] CrysAlis CCD, Oxford Diffraction Ltd. Abingdon, England, 2002.
- [22] CrysAlis RED, Oxford Diffraction Ltd. Abingdon, England, 2002.
- [23] G.M. Sheldrick, A short history of SHELX, *Acta Cryst. A* 64 (2008) 112–122.
- [24] G.M. Sheldrick, New features added to the refinement program SHELXL since 2008 are described and explained, *Acta Cryst. C* 71 (2015) 3–8.
- [25] C.F. Macrae, I.J. Bruno, J.A. Chisholm, P.R. Edgington, P. McCabe, E. Pidcock, L. Rodriguez-Monge, R. Taylor, J. van de Streek, P.A. Wood, Mercury CSD 2.0 - new features for the visualization and investigation of crystal structures, *J. Appl. Crystallogr.* 41 (2008) 466–470.