Synthesis, spectral Characterization and studies of Some Divalent Metal Ion Complexes with Ligand of [3-(1H-indol-3-yl)-2-(3-(4-methoxybenzoyl)thioureido)propanoic acid] (MTP)

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Abstract

In this work, prepared new ligand[3- (1H-indol-3-yl) -2- (3-(4-methoxybenzoyl)thioureido) propanoic acid](MTP) has been synthesized by reaction of 4-Methoxybenzoyl isothiocyanate with tryptophane(1:1). The ligand was characterized by elemental microanalysis C,H,N,S, FT-IR, UV-Vis and ¹H,¹³C NMR spectra, Some transition metals complexes of this ligand were prepared and characterized by FT-IR, UV-Vis spectra, conductivity measurements, magnetic susceptibility and atomic absorption, From obtained results the molecular formula of all prepared complexes were [M(MTP)₂] (M⁺² =Mn, Co, Ni, Cu, Zn, Cd and Hg), the proposed geometrical structure for all complexes were tetrahedral except copper complex has a square planer geometry around metallic ion with bidentate ligand(MTP).

Key Words: Tryptophane, 4-Methoxybenzoylisothiocyanate, Divalent metal ion, Complexes.

1. Introduction

α-Tryptophan was reported to inhibit the growth of Bacterium coli[1] and to be used as IDO inhibitors[2]. In addition,tryptophan analogues are important building blocks for the synthesis of biologically active compounds ranging from peptide mimetics to natural products[3]. Among various synthetic methods, two strategies have been used for the preparation of tryptophan analogues. The metal complexes derived from amino acid and their derivatives have important pharmaceuticals applications, they have been exploited in cancer multi drugs resistance [4] tested as anti-material[5] and exhibit anti-tumor activity [6]. Metal complexes are gaining importance in recent years particularly in the design of repository, slow release or long acting drug in nutrition and in the study of metabolism [7]. Metal ions are known to accelerate the drug action [8]. The transition metal complexes of tryptophan have been extensively examined due to their wide applications in various fields like
biological, pharmacological, clinical, analytical and industrial area. These ligands have been playing an important part in the development of coordination chemistry as they easily form stable complexes with most of the transition metal ions[9]. The interaction of these donors and metal ion give complexes of different geometries and these complexes are potentially biologically active[10].

Complexes of transition metals containing ligands with N,O donors play an essential role in agricultural, pharmaceutical, industrial chemistry[11-12], antifungal, antibacterial, anticancer and catalytic activities[13-14]. T. Kavitha and Co Worker reported synthesis Schiff base from tryptophan with napthylidene-4-aminoantipyrine and also their metal complexes and have the general formal[M] where M= VO(II), Co(II), Ni(II), Cu(II) and Zn(II)[15]. Also synthesis, characterization and toxicity studies of Schiff based driven from pyridine carboxaldehydes and tryptophan and their metal complexes[16].

The aim of the present study was the synthesis, characterization of Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) complexes derived from tryptophan.

2. Experimental

2.1. Material and measurements

All reagents were analar or chemical pure grade by Sigma-Aldrich, BDH, Merck and Fluka. The chemical tryptophane, Ammonium thiocyanate, 4-Methoxybenzoylchloride, metal chloride salts (MnCl2.4H2O), (CoCl2.6H2O), (NiCl2.6H2O), (CuCl2.2H2O), (ZnCl2), (CdCl2.2H2O) and all solvents were used without purification.

1H and 13C–NMR was recorded using Ultra Shield 300 MHz Swissarl and at University of Al al-Bayt, Jordan. Melting point was recorded by using Stuart- melting point apparatus. FT-IR spectra were recorded as KBr disc using 3800 Shimadzu in the range of (4000-400) cm⁻¹. Electronic spectra were obtained using UV-160 Shimadzu spectrophotometer at room temperature for 10⁻³M solution DMSO with 1.000 ± 0.001 cm matched quartz cell. Molar Conductivity was measured at 25°C for 10⁻³M solution of DMSO by using Philips PW.Digital. Elemental micro analyses(C.H.N.S) were performed using Acrlo Erba 1106 elemental analyzer. Magnetic susceptibility measurements were obtained by balance magnetic susceptibility by model MSB-MKL. Metal contents of the complexes were determined by atomic absorption technique by using Shimadzu (AA680G).

2.2. Preparation of ligand(MTP)

The ligand was prepared by two steps

Step 1. Preparation of the (4-MethoxyBenzoyl isothiocyanate).[17]

Mixture of ammonium thiocyanate (2g, 26mmol) and 4-methoxybenzoyl chloride (3.55ml,26mmol) in (25ml) of acetone was stirred under refluxed for 3hrs and then filtered, the filtrate was used for further reaction.

Step 2. Preparation of 3-(1H-indol-3-yl)-2-(3-(4-methoxybenzoyl) thioureido) propanoic acid (MTP)

(5.31 g, 26mmol) of tryptophane in (20ml) ethanol was rapidly added to 4-methoxybenzoyl isothiocyanate and maintaining reflux. After refluxing for 6hrs, the resulting solid was collected, washed with acetone and recrystallization from ethanol, Yield(%82), (M.P=250-252)°C, C% found (60.50) calc.(60.45), H% found (4.67), calc.(4.78),N% found (10.40),
calc.(10.57), S% found (8.45), calc.(8.06) (scheme:1).

Scheme (1) Preparation of ligand(MTP)

2.3. Synthesis ligand (MTP) complexes

The complexes [M(MTP)2] have been prepared by the reaction of (0.794g, 2mmol) of ligand(MTP) in (10ml) ethanol containing (0.12gm, 2mmol) of KOH with (1mmol) off metal chloride (0.20gm, 0.237gm, 0.170gm, 0.237gm, 0.136gm, 0.20gm and 0.272gm for MnCl2.4H2O, CoCl2.6H2O, CuCl2.2H2O, NiCl2.6H2O, ZnCl2, CdCl2.2H2O and HgCl2) respectively dissolved in (20ml) absolute ethanol and refluxed with stirring under for 3 hours at room temperature, the precipitate was collected by filtration, washed with(1:1) mixture of water: ethanol and dried in an oven(50°C). Table(1) exhibit some physical properties of the prepared complexes.

3. Results and Discussion

Ligand and complexes were identified on the basis of elemental analysis and spectral studies. color, yield and elemental analysis data are represented in Table (1).

3.1. Ligand (MTP)

1H-NMR spectrum of the ligand (MTP) fig.(1)which was recorded in CDCl3 as a solvent showed the following signals: singlet at δ(1.419 ppm for (H, CH), singlet at δ (3.902 ppm for (3H, OCH3), singlet at δ (4.663-4.714)ppm for (2H, CH2), multiplet at spreaks at δ (6.985-7.015)ppm for(4H, aromatic protons), singlet at δ(7.184)ppm for(1H, NH Sec amine), multiplet peaks at δ(7.776-7.852)ppm for (4H, indolring), singlet at δ (10.486)ppm for (1H, COOH).

13C-NMR spectrum of the ligand (MTP) fig.(2)which was recorded in CDCl3 as a solvent showed the following signals : single peak at δ(13.260) ppm for (CH2), single peak at δ (55.630 )ppm for (OCH3), single peak at δ (70.234)ppm for (CH), single peak at δ (111.878-123.511)ppm for(aromatic carbons), single at δ(130.534-130.155)ppm for(carbon in pyrloing), singlet at δ(160.718-159.586)ppm for (carbon in benzening), single peak at δ (164.409)ppm for (C=O sec.amide), single peak at δ (164.849)ppm for (COOH), single peak at δ (189.047)ppm for(C=S) [18-19].

The UV-Vis spectrum of the free ligand (MTP). Fig.(5) exhibits a high intense absorption peaks at (37313)cm -1 and (33222) which way attributed to electronic transitions type π π* and π π*[20,21]. The data of electronic spectrum of the free ligand (MTP) were listed in table(3).

3.2. Complexes of the ligand (MTP)

All complexes soluble in some common solvent such as DMF, DMSO and relatively thermally stable. The molar conductivity values of all complexes in DMSO solvent in 10^-3M at 25°C were recorded in rang (4-12) Ohm -1 cm² mol⁻¹ indicated non-electrolytic(table-1)[22]. The atomic absorption measurements for all complexes gave approximated values when its comparison with theoretical values, Table(1) includes the physical
properties for the ligand and its complexes.

3.3. FT-IR Spectra

These spectra exhibited marked difference between bands Fig. (4) belonging to the stretching vibration of $\nu$(COO)$^\text{sym}$ in the range (1354-1465) cm$^{-1}$ shifted higher frequencies by (51-162) cm$^{-1}$ and $\nu$(COO)$^\text{asym}$ (1581-1695) cm$^{-1}$ suggesting shifted lower frequencies by (135-31) cm$^{-1}$ of the possibility of the coordination of ligand through the oxygen atom at the carbonyl group[23], while the band caused by $\nu$(N-H) appeared between (3317-3390) cm$^{-1}$ shifted to lower frequencies by (85-12) cm$^{-1}$ which indicates to the coordination of ligand through the nitrogen atom at $\nu$(N-H)group[24]. The stretching vibration band $\nu$(C=O amido) and $\nu$(C=S) either show no change or very little in their frequencies (1604) cm$^{-1}$ and (1253-1261) cm$^{-1}$ respectively there for indicating do not coordinate to the metal ion. Metal-oxygen and metal-nitrogen bonds were confirmed by the presence of the stretching vibration of $\nu$(M-O) and $\nu$(M-N) around (452-520) cm$^{-1}$ and (428-470) cm$^{-1}$ respectively[25]. Table(2) describe the important bands and assignment for all prepared complexes and Fig.(4): Infrared spectrum of one from prepared complexes [Cd(MTP)$_2$].

3.4. Magnetic moment

The values of observed magnetic moments at room temperature and effective magnetic moment ($\mu_{\text{eff}}$) for the Mn(II), Co(II), Ni(II) and Cu(II) complexes are shown in table(1). Mn(II), Co(II), Ni(II) and Cu(II) complexes exhibit $\mu_{\text{eff}}$ (5.92, 4.82, 2.82 and 1.74) B.M respectively. These values are typical for high spin tetrahedral and squar planer complex for Cu(II) ion geometry coordinated around metale complexes.[26]

3.5. Electronic spectra of complexes

3.5.1 [Mn(MTP)$_2$]d$^5$

The complex of Mn(II) Fig.(6) shows band at (37174) cm$^{-1}$ due to (L.F) and other bands at (30393) cm$^{-1}$ and (21505) cm$^{-1}$ which are caused by the charge electronic transfer (C.T) and $^4$A$_1$ $\rightarrow$ $^4$T$_{1(G)}$, respectively, suggesting tetrahedral geometry around Mn(II) ion[27].

3.5.2 [Co(MTP)$_2$]d$^7$

The spectrum of the complex gave four bands at (37174) cm$^{-1}$, (29239) cm$^{-1}$, (17090) cm$^{-1}$ and (12048) cm$^{-1}$ attributed to (L.F) and C.T$_{\text{mix}}$ with $^4$A$_2(F)$ $\rightarrow$ $^4$T$_{1(P)}$, $^4$A$_2(F)$ $\rightarrow$ $^4$T$_{1(F)}$ and $^4$A$_2(F)$ $\rightarrow$ $^4$T$_{2(F)}$ respectively and the rachinter electronic repulsion parameter (B) was found to be (679) cm$^{-1}$, from the relation $\beta=B/B'$. [$\beta$ was found to be equal(0.70), these parameter are accepted to Co(II) tetrahedral complex[28].

3.5.3 [Ni(MTP)$_2$]d$^8$

The spectrum of complex of Ni(II) has revealed the following (L.F) and C.T, $^3$T$_{1(F)}$ $\rightarrow$ $^3$T$_{1(P)}$, $^3$T$_{1(F)}$, $^3$A$_2$ and $^3$T$_{1(F)}$ $\rightarrow$ $^3$T$_{2(F)}$ transition at (37174) cm$^{-1}$, (30303) cm$^{-1}$, (25641) cm$^{-1}$, (18867) cm$^{-1}$ and (10989) cm$^{-1}$ respectively, the(B') value found to be (769.4) cm$^{-1}$, while $\beta$ was equal to (0.739) these are the characteristics for tetrahedral complexes of Ni(II).[29,30]

3.5.4 [Cu(MTP)$_2$]d$^9$

The spectrum of complex of Cu(II) show four bands at (37174) cm$^{-1}$, (30303) cm$^{-1}$, (15267) cm$^{-1}$ and (13315) cm$^{-1}$ caused to (L.F), (L.F), $^2$B$_{1g}$ $\rightarrow$ $^2$A$_{1g}$, $^2$B$_{1g}$ $\rightarrow$ $^2$B$_{2g}$ transition respectively, which was a good agreement for squar planer complex for Cu(II) ion fig.(8)[31,32].
3.5.5 [Zn(MTP)₂], [Cd(MTP)₂] and [Hg(MTP)]

Shows only charge transfer (C.T) and (L.F) of (M→L) in range 22675-31645 cm⁻¹ and (28985-37174) respectively [33,34]. All transition with their assignments are summarized in Table (3).

4. Conclusions

New ligand (MTP) and their metal complexes were prepared and characterized by spectral and analytical data. The synthesized ligand act as bidentate, coordinating via oxygen and nitrogen atoms of (COO⁻) and (N-H) groups respectively as illustration fig.(7). Magnetic and electronic studies reveal "tetrahedral" structure for all complexes except Copper complex has a "square planer" geometry.

References

triphenylphosphine / triphenylarsine and tetradehtenate Schiff bases Inorg.

<table>
<thead>
<tr>
<th>Compound</th>
<th>M.wt (gm/mole)</th>
<th>Color</th>
<th>M.p°C</th>
<th>M% Calculation (Found)</th>
<th>Molar Cond. Ohm⁻¹ cm² Mol⁻¹ in DMSO</th>
<th>μeff (B.M)</th>
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<tbody>
<tr>
<td>Ligand (MTP)</td>
<td>397</td>
<td>Brown</td>
<td>250-252°C</td>
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<td>2</td>
<td>–</td>
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<tr>
<td>[Mn(MTP)₂]</td>
<td>846.94</td>
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<td>115-117°C</td>
<td>6.49 (6.19)</td>
<td>12</td>
<td>5.92</td>
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<td>850.93</td>
<td>Blue</td>
<td>158-160°C</td>
<td>6.93 (6.82)</td>
<td>11</td>
<td>4.82</td>
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<td>[Ni(MTP)₂]</td>
<td>850.71</td>
<td>Green</td>
<td>165-168°C</td>
<td>6.93 (6.85)</td>
<td>9</td>
<td>2.82</td>
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<td>Deep Brown</td>
<td>105-108°C</td>
<td>7.42 (7.33)</td>
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<td>1.74</td>
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<td>181-183°C</td>
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<td>Deep Yellow</td>
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<td>[Hg(MTP)₂]</td>
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<td>113-115°C</td>
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Table No. (1): Some physical properties of the ligand (MTP) and its complexes

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<th>Compounds</th>
<th>$u$(O−H)</th>
<th>$u$(N−H)</th>
<th>$u$(COO) asym</th>
<th>$u$(COO) sym</th>
<th>$\Delta u$ (COO)</th>
<th>$u$(C=O) Amide</th>
<th>$u$ (C=N)</th>
<th>$\nu$ (M-O)</th>
<th>$\nu$ (M-N)</th>
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</thead>
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<tr>
<td>Ligand MTP</td>
<td>3078(m)</td>
<td>3402(m)</td>
<td>1716(m)</td>
<td>1303(w)</td>
<td>413</td>
<td>1604(s)</td>
<td>1211(s)</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>[Mn(MTP)$_2$]</td>
<td>3348(b)</td>
<td></td>
<td>1654(s)</td>
<td>1419(s)</td>
<td>235</td>
<td>1604(s)</td>
<td>1211(m)</td>
<td>478(w)</td>
<td>428(w)</td>
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<td></td>
<td>1581(s)</td>
<td>1354(s)</td>
<td>227</td>
<td>1604(s)</td>
<td>1257(m)</td>
<td>479(w)</td>
<td>457(w)</td>
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<td></td>
<td>1581(s)</td>
<td>1373(s)</td>
<td>208</td>
<td>1604(w)</td>
<td>1257(w)</td>
<td>462(w)</td>
<td>432(w)</td>
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<td>3387(b)</td>
<td></td>
<td>1666(s)</td>
<td>1435(s)</td>
<td>231</td>
<td>1604(w)</td>
<td>1257(m)</td>
<td>452(w)</td>
<td>447(w)</td>
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<td>1423(s)</td>
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<td>1604(s)</td>
<td>1211(s)</td>
<td>478(w)</td>
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<td>1396(s)</td>
<td>266</td>
<td>1604(S)</td>
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<tr>
<td>[Hg(MTP)$_2$]</td>
<td>3390(b)</td>
<td></td>
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<td>1465(s)</td>
<td>230</td>
<td>1604(S)</td>
<td>1253(w)</td>
<td>520(w)</td>
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Table (2): The characteristic infrared band in cm$^{-1}$ for free ligand (MTP) and its metal complexes

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<th>Compounds</th>
<th>$\lambda$(nm)</th>
<th>$\nu$ (cm$^{-1}$)</th>
<th>$\nu$ as sym</th>
<th>$\nu$ sym</th>
<th>$\Delta \nu$ (COO)</th>
<th>$\nu$ (C=O)</th>
<th>$\nu$ (C=N)</th>
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<td>37313</td>
<td>1.272</td>
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<td>1272</td>
<td>927</td>
<td></td>
<td></td>
<td></td>
<td>$\pi\rightarrow\pi^*$</td>
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<tr>
<td></td>
<td>301</td>
<td>33222</td>
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<td>1272</td>
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<td></td>
<td></td>
<td>$n\rightarrow\pi^*$</td>
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<tr>
<td></td>
<td>269</td>
<td>37174</td>
<td>1.490</td>
<td>28</td>
<td>1490</td>
<td></td>
<td></td>
<td></td>
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<td>L.F.</td>
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<td>[Mn(MTP)$_2$]</td>
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<td>37174</td>
<td>1.525</td>
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<td></td>
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<tr>
<td></td>
<td>342</td>
<td>29239</td>
<td>2.041</td>
<td>28</td>
<td>2041</td>
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<td></td>
<td></td>
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<td>C.T Mix whit $^{4}A_2\rightarrow$</td>
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<td>L.F.</td>
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<td>[Hg(MTP)$_2$]</td>
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<td>$^{2}B_1g\rightarrow^{2}A_1g$</td>
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Table (3) Electronic spectral data of ligand (MTP) and its complexes in DMSO solvent
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<td>L.F</td>
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<td>345</td>
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</tr>
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<td></td>
<td>357</td>
<td>28011</td>
<td>1.170</td>
<td>1170</td>
<td>C.T</td>
</tr>
</tbody>
</table>

L.F = ligand field, C.T = charge transfer

Figure No. (1): $^1$H-NMR spectrum of ligand (MTP)

Figure No. (2): $^{13}$C-NMR spectrum of ligand (MTP)

Figure No. (3): Infrared spectrum of ligand (MTP)

Figure No. (4): Infrared spectrum of complex [Cd(MTP)$_2$]
Figure No. (5): U.V spectrum of ligand (MTP)

Figure No. (6): U.V spectrum of complex [Ni(MTP)_2]

Figure No. (7): The proposed tetrahedral structure of the complexes [M(MTP)_2].

Figure No. (8): The proposed squar planer structure complex for [Cu(MTP)_2].

كحضري تشخيص طيفي ودراسة بعض المعقدات الفلزية ثنائية التكافؤ مع الليكانت (3-هيدروجين 1-أندول-2-3)-4-ميثوكسي بنزويل(ثابوريدو) حاضم بروبانويك

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الخلاصة:
في هذا العمل حضر الليكانت الجديد [3-(هيدروجين 1-أندول-2-3)-(4-ميثوكسي بنزويل(ثابوريدو) حاضم بروبانويك] (MTP) وذلك من مفاعله (4-ميثوكسي بنزويل آيزوترويلبانويل) مع الحاضم الأميني التربوتان ونسبة (1:1) وشخص بوساطة التحليل الدقيق لمعادل (C.H.N.S) والأشعة تحت الحمراء والأشعة فوق البنفسجية. الدراسة تطفل تربوتان النووي الفلزية، كما حضرت وشخصت معادل أملاح (MTP) مع الليكانت (Hg, Cd, Zn, Cu, Ni, Co, Mn) بعض عوامل العناصر الانتقالية الثلاثية التكافؤ وشخصت معادل معادل الحمض المحمية باستخدام الأشعة تحت الحمراء والأشعة فوق البنفسجية - المرمية والتوصيلية المولارية والحساسية الفلزية والاستنتاجات التي واستنتج إن المعادلة لها شكل رياضي سطح ماعدا معقد التحصين له شكل مربع مستوي حول الينون الفلزية مع الليكانت (MTP) ثنائي السنت الكلمات المفتاحة: تربوتان، 4-ميثوكسي بنزويل آيزوترويلبانويل، أيونات فلزية ثنائية التكافؤ، معادلة.