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Synthesis and Characterization of Some 5-Bromouracil Metal ion Complexes

Said M. Teleb, Mohamed G. Abdel Wahed, Ahmed F. El Farargy, Marwa I.Y. Ahmed
Chemistry Department, Faculty of Science, Zagazig University, Zagazig, Egypt
Corresponding author:mmr656776@gmail.com

ABSTRACT: ***Five new complexes [Mg(BrU)2].8H2O (A), [Sr(BrU)2.(H2O) 2] (B), [Ca(BrU)2(H2O)2].H2O (C), [Pb(BrU)2] (D) and [Hg(BrU)2] (E) were prepared by the reaction of 5-bromoouracil ligand with MgCl2.6H2O, SrCl2.6H2O, CaCl2.2H2O, Pb(NO3)2 and HgCl2 salts, respectively. The metal complexes were characterized by conventional methods through their melting point, molar conductivity, elemental analysis, IR, UV- Vis., 1 H-NMR analyses. The obtained data indicated that the ligand***
undergoes keto ↔ enol tautomerism and behaves as mononegative bidentate donor and interacts with all metal ions. Thermal analysis indicate that the complexes degrade more or less, through three main steps representing the dehydration of physically absorbed and coordinated water molecules then the decomposition of the unhydrous species leading to metal oxides as final products. Some of the prepared complexes were tested against gram positive and gram negative bacteria and fungi which showed effective activity.

KEYWORDS: 5-Bromoouracil–M^{2+} Complexes, IR, Thermal Analyses, ^{1}H NMR, antimicrobial and antifungal activities.

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I. INTRODUCTION

5-bromouracil Figure (1) is an uncommon nucleotide bases which have the ability to coordinate with metal [1-4] or to bind to tissues through metals [5]. As 5-Bromouracil is closely similar to thymine, it is readily introduced into DNA during its reproduction (as 5- bromouridylic acid) in the location which typically occupied by thymine [6]. 5- bromouracil and its metal complexes can be used in pharmacological applications as anti-microbial [5,7] and anti-tumour agents [1].

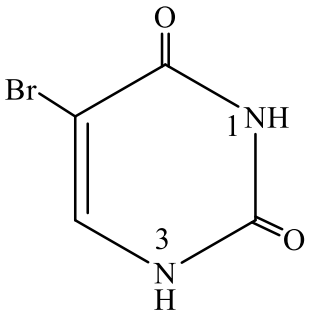


Figure (1): The structure of uracil

Furthermore, uracil and halo-uracil complexes can be used as catalyst in various ester hydrolysis which inhibits Dalton's Lymphoma tumour system [8] which can be conclude DNA replication in viruses and other cell culture systems [9,12]. Uracil is crystalline organic compound of the pyrimidine family that present paring with adenine nucleobase through hydrogen bond as a component of ribonucleic acid (RNA), colorless, a planar, unsaturated molecule that has the ability to absorb light [13]. 5-Bromouracil thermal decomposition is very dangerous, because it emits very toxic fumes, hydrogen bromide and nitrogen oxide, through its decomposition process [14].

II. Materials and Methods

II.1. Chemicals and reagent

All chemicals used are of analytical grade and used without further purifications. 5-Bromouracil was purchased from sigma-Aldrich chemical company, $MgCl_2.6H_2O$, $SrCl_2.6H_2O$, $CaCl_2.2H_2O$, $Pb(NO_3)_2$, $HgCl_2$ and NaoH were purchased from Fluka Chemical Company.

II.2. Synthesis of ligand and its metal complexes

II.2.1. Synthesis of 5-bromouracil ligand solution

2 mmol of 5-bromouracil (0.9 g) was suspended in about 30ml of distilled water, (initial pH = 4.2) and then a NaOH solution was added drop wisely until finally reaching pH=10 or till complete dissolution of ligand.

II.2.2. Synthesis of metal complexes

The metal complexes of 5-bromouracil named $[Mg(BrU)_2].8H_2O$, $[Sr(BrU)_2.(H_2O)_2]$, $[Ca(BrU)_2(H_2O)_2].H_2O$, $[Pb(BrU)_2]$ and $[Hg(BrU)_2]$ were prepared by mixing the prepared ligand solution with an aqueous solution (30ml) of the corresponding metal salts, (1 mmol) $MgCl_2.6H_2O$ (0.406g), $SrCl_2.6H_2O$ (0.532g), $CaCl_2.2H_2O$ (0.294g), $Pb(NO_3)_2$ (0.662g) and $HgCl_2$ (0.542g) in general molar ratio 1:2 , metal salt: ligand, respectively. The reaction mixture was stirred at room temperature for 24h and then left for slow evaporation at room temperature until lose one third of its volume. The formed precipitate was filtered off, washed several times with few drops of distilled water and then dried in a vacuum dissector over anhydrous $CaCl_2$

II.3. Instrumentation

The synthesized ligand and its metal complexes were characterized by their elemental analysis CHN contents were carried out in the Microanalysis Department of Cairo University, Cairo, Egypt using a VARIOEL III elemental analyzer. Infrared measurements (KBr-pellets) were carried out using a Unicom sp1000 IR spectrophotometer at Cairo University. Uv spectra were measured in the region of 800-200nm using UV-3101PC Shimadzu (micro analytical center at Cairo University). ^{1}H-NMR spectra were recorded on Varian Mercury VX-300 NMR Spectrometer using DMSO-d^6 as a solvent and TMS as internal reference (micro analytical center at Cairo University). Molar conductivities of freshly prepared solutions (10^{-3} M) in DMSO at room temperature were measured using CONSORT K410 conductivity meter. Thermogravimetric (TGA) and differential thermogravimetric analyses (DTG) were carried out under N_2 atmosphere (30 ml/min.) using detectors model TGA-50H Shimadzu thermal analyzer. The rate of heating of the sample was kept at 10 °C/min (micro analytical center at Cairo University). The metal contents of the prepared complexes are detected by volumetric quantitative analysis (titration with EDTA in the presence of a suitable indication) or by atomic absorption technique using the corresponding lamp.

Table 1: **Elemental analysis and physico-analytical data for 5-Bromouracil and its metal complexes.**

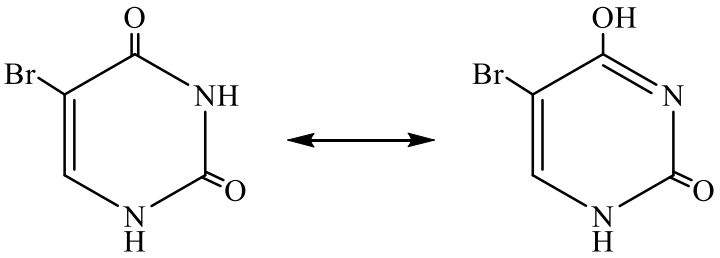
Compounds	M.wt (gm/mol)	Mp /oC	Color	Found (Calcd.) (%)				Λ S cm^2 mol^{-1}
				C	H	N	M	
5-Bromouracil $[C_4H_3BrN_2O_2]$	190.98	310	off-white	25.16 (24.81)	1.58 (1.67)	14.67 (14.32)	_	
$[C_8H_4Br_2N_4O_4Mg]8H_2O$	545.93	310	White	21.92 (22.03)	1.84 (1.99)	12.79 (13.070	5.48 (5.66)	22.5
$[C_8H_4Br_2N_4O_4Sr]2H_2O$	503.60	355	White	19.13 (20.04)	1.61 (1.91)	11.16 (12.11)	17.52 (17.08)	28.51
$[C_8H_4Br_2N_4O_4Ca]3H_2O$	474.81	280	Gray-white	21.15 (20.86)	1.78 (1.88)	12.34 (12.58)	8.81 (8.45)	13.42
$[C_8H_4Br_2N_4O_4Pb]$	587.15	260	Orange	15.44 (16.01)	1.30 (1.66)	9.01 (9.44)	33.44 (33.03)	7.66
$[C_8H_4Br_2N_4O_4Hg]$	579.85	300	Orange	15.59 (16.23)	1.31 (1.55)	9.10 (8.88)	32.80 (32.42)	11.51

III. RESLUTS AND DISCUSSIONS

II.1. Infrared Spectra of ligand and its metal complexes

The IR spectra for the synthesized ligand and its metal complexes are given in Figure (5) and tentative assignments for the well-characterized vibrations are given in Table (1).

The spectrum of the free 5-bromouracil reveals two absorption bands at 3360 and 3168 cm^{-1} which may be assigned to ν(N3-H) and ν(N1-H) bond vibrations, respectively [15]. The band due to ν(N3-H) bond vibration disappears in the spectra of all metal complexes indicating that N3 atom is deprotonated and shared in bonding with the metal ions. The IR spectrum of the free ligand also shows a very strong and broad band at 1677 cm^{-1} characteristic to (C=O) carbonyl bond vibration [16]. The broadness of this band may be attributed due to the expected overlap of two distinct vibrations of (C4=O) and (C2=O) bonds vibrations. IR spectra of metal complexes show a strong band at 1631, 1644, 1629, 1634 and 1621 cm^{-1} characteristic to (C=O) bond vibration of $[Mg(BrU)_2].8H_2O$, [Sr(BrU)2.(H2O)2], [Ca(BrU)2(H2O)2].H_2O, $[Pb(BrU)_2]$ and $[Hg(BrU)_2]$ complexes, respectively. The lower frequency shift (35-58 cm^{-1}) indicates the coordination of the carbonyl group to the metal ions. The spectra of the metal complexes reveal a new absorption band at 1562, 1603, 1562, 1580 and 1526 cm^{-1} belong to ν(C=N) bond vibrations for [Mg(BrU)2].8H2O, [Sr(BrU)2.(H2O)2], [Ca(BrU)2(H2O)2].H_2O, [Pb(BrU)2] and [Hg(BrU)2]metal complexes, respectively [17]. This could be understood if we proposed a structural transformation of the ligand from keto to enol form as a consequence of the complexation structure (II). This coordination mode of the ligand is greatly supported by observing a week band in the spectrum of metal complexes in the regions 700-500 cm^{-1} attributed due to M-N and M-O bonds stretching vibrations, respectively [18].



Keto-enol tautomerism

Figure (2): The ionized form of uracil.

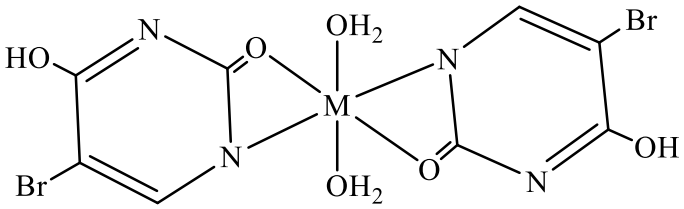
III.2. Molar Conductivity

The molar conductivities were measured in at room temperature in DMF solutions (10^{-3} M solution) for the free ligand and its metal complexes. The metal complexes have molar conductivities (ΛM) of 3 - 30 S cm^2 / mol and are therefore nonelectrolytes which agree quite with the proposed formulas of the complexes[19].

III.3. Electronic Spectra

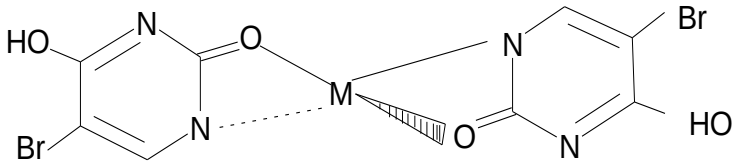
The electronic spectra of the free 5-bromouracil and its metal complexes are given in Figure (6) and the assignments of the absorption bands are given in Table (2). The spectrum of the free 5-bromouracil ligand exhibits two main electronic transitions at 304 and 346 nm. The former one which may be assigned due to π-π* transition [20] is also observed in the spectra of the complexes but with red shift to longer wave length in the range of 14-20 nm. This could be understood on the bases of the electronic change in the uracil nucleus as a consequence of the coordination with the metal ions. The latter band of 346 nm in the free ligand disappeared in the spectra of the complexes. Therefore, we can conclude that, the transition may be associated with n- π* of the group (C=O) of the uracil which is not present anymore, because of the proposed coordination with the metal ions and the transformation of the ligand from the keto to enol form. According to the foregoing discussion and the proposed molecular formulas of the complexes, Hg^{2+} and Pb^{2+} ions interact with two molecules of 5-bromouracil giving a complexes of a tetrahedral geometry figure (3) with the ligand as mononegative bidentate through one oxygen of the carbonyl group and N3 atom. On the other hand, the complexes are Sr, Ca and Mg are of octahedral geometry figure (4) with two water molecules above and below the

plane containing the oxygen and nitrogen atoms on the other hand the complexes are Sr, Ca and Hg are of octahedral geometry scheme (1) with two water molecule above and below the plane containing the oxygen and nitrogen atoms.



M= Ca, Sr

Figure (3): The proposed structures of uracil-Ca or Sr complexes.



M = Mg, Hg, Pb

Figure (4): The proposed structures of uracil-Mg, H or Pb complexes.

III.4. ^{1}H-NMR spectra

The ^{1}H-NMR spectra of the free 5-bromouracil ligand, the [Hg(BrU)2] and [Ca(BrU)2.(H2O)$_2$].H$_2$O complexes are given in Figure (7), while the assignments of the observed signals are given in Table (3). The data obtained from the ^{1}HNMR spectra greatly support our conclusion on the coordination mode of 5-bromouracil. The up- field shift of C6-H in the free ligand from 7.88 ppm to 7.74 and 7.73 ppm in Hg and Ca-complexes, respectively, is strongly related to the increased aromaticity in the uracil ring due to the keto to enol transformation. The same conclusion can be also supported by observations that, the two signals due to N1-H and N3-H appeared in the spectrum of free 5-bromouracil are disappeared in the complexes spectra and instead a signal at 11.25 and 11.28 ppm in the spectra of [Hg(BrU)2] and [Ca(BrU)2.(H2O)$_2$].H$_2$O, respectively ,is observed . This signal may be assigned due to OH proton in good consistency with the proposed coordination manar of 5-bromouracil with these metal ions.

III.5. Thermal analysis

Thermogravimetric (TGA) and differential thermo-gravimetric (DTA) analyses were carried out within the temperature range of 25 – 800^{o}C with heating rate 10^{o}C/min under the flow of nitrogen gas as atmospheric. Representative thermograms are given in Figure (8) and data obtained are recorded in table(5),where the maximum temperature, T_{max}, values of the decomposition steps along with the corresponding weight loss are given.

The thermogram of the free ligand (5-Bromouracile) shows as expected complete decomposition in one step at T_{max} of 320°C indicating its pure organic nature.Magnesium complex, [Mg(BrU)2]8H2O thermogram shows two stages of decomposition. The first stage occurs at temperature maximum of 125^{o}C and may be corresponding to the loss of the crystalline water (8H$_2$O) the calculated value 26.73%

Consists with the found value of 25.18% for the loss of 8 water molecules. The second stage of decomposition distributed among two maxima 327 and 450 ^{o}C corresponding to the loss of organic moiety. The data show that MgO may be the decomposition residue where the found value of 6.74% agree quite well with the calculated value of 7.32%. [Sr(BrU)2.(H2O)$_2$], [Ca(BrU)2(H2O)2]H$_2$O thermograms exhibit decomposition step at 190 and 225 ^{o}C corresponding to loss in weight of (8.04) and (8.74), respectively. The loss of two coordinated water molecules at such higher temperature is expected and the loss values agree quite well with the calculated value of (7.14) and (7.57) for the two complexes respectively. The calcium complex shows another step of decomposition at lower

temperature 130 ^{o}C corresponding to the loss of one uncoordinated water molecule, where the found and calculated weight loss are in constituency.

The two complexes exhibit a stage of decomposition at temperature maxima of 373 and 421 ^{o}C corresponding to the loss of organic moieties of the complexes. The calculated values agree well with the bond values of the weight loss, see Table(5).

Finally, the lead complex, [Pb(Bru)$_2$], thermogram shows one stage of decomposition corresponding to its proposed formula. This stage occurs at 400 ^{o}C and associated with a weight loss of (58.16) due to the loss of the organic part of the complex. The decomposition residue of 42.36% consistent well with PbO_2 as a final decomposition product, where the calculated value of 40.70% consists with the found one. The gravimetric analysis of [Hg(BrU)$_2$] complex does not show any loss of weight in the range of temperature up to 250 ^{o}C consistent with for proposed formula.

III.6. Biological activity

The antimicrobial and antifungal activities of selected representative complexes were tested against two bacterial strains; Staph.aureus, E.coli and two fungi; Asper.ocherations, Asper.niger species. Standard drug; Ampicillin and DMF solvent control were screened separately for their antibacterial activities as positive and negative controls [23,24] respectively. The results are cited in Table 6 shown graphically in terms of inhibition zone and percent activity indexes in Fig (9) and (10).

Inspection of the biological and antifungal data suggests that the three uncomplixed drugs showed comparable efficiencies nearly close to those of ampicillin taken as positive control. The studied complexes exhibit a high activity against the tested organisms compared to Ampicillin. Also, percent Activity Index data show that for E. coli. Mg (II) complex has the higher activity than Mg complex, the same is observed in case of Stph.aureus. On the other hand, for the studied fungi organism the activity of Hg(II) is higher than that of Mg(II) complex. For all cases, the activities of the complexes are slightly lower than those of the parent drugs.

Table 2: Infrared frequencies (cm^{-1}) and tentative assignments for 5-Bromouracil, [Mg(BrU)2].8H2O (A), [Sr(BrU)2.(H2O)2] (B), [Ca(BrU)2(H2O)2].H$_2$O (C), [Pb(BrU)2] (D) and [Hg(BrU)2] (E).

Assig.	ν(O-H), H2O	ν(N3-H)	ν(N1-H)	ν(C-H) aromatic	ν(C=O)	ν(C=N)	υ(C–C) δ (N1–H)	υ(C–N) υ(C–O)	ν(C-Br)	ν(M-N)	ν(M-O)
Br-U-H	3467w	3360s	3168m	3060 m	1679 vs	-	1427s	1224m	1054 w	-	-
A	3376 s	-	3121w	3121 s	1631s	1591s	1465W	1272w	1071w	788m 648m	-
B	3442w	-	3121m	3047m	1644m	1603s	1488vs	1290w	1057w	786w 759w 673w	608w
C	3499m	-	3154w	3102 m	1629 vs	1562 m	1474m	1291w	1007w	780m 744w 659s	461w
D	3434w	-	3145w	3055w	1621s	1526s	1438s	1225w	1003w	766m 670w 644w	580w 546w
E	3412m	-	3166w	3055w	1634s	1580s	1431m	1285m	1081w	766m 711w 647w	538w 587w

where s=strong, w=weak, v=very strong, m=medium, sh=shoulder, v=stretching.

Table 3: UV-Vis. spectra of 5-Bromouracil and its metal complexes (200-800 nm).

Assignments (nm)	(Br-U-H)	*Mg*(II)	*Sr*(II)	*Ca*(II)	*Hg*(II)	*Pb*(II)
π-π* transitions	304	320	318	318	316	314
n-π* transitions	346	-	-	-	-	-

Table 4: ^{1}H NMR signals (ppm) and their assignments.

Compound	*C6-H*	*N1-H*	*N3-H*	*OH*
5-Bromouracil	7.88(1H)	11.13(1H)	(11.52)(1H)	---
[Ca(BrU)2(H2O)2]	7.78(1H)	-	-	11.28(1H)
[Hg(BrU)2]	7.74(1H)	-	-	11.25(1H)

Table 5: The maximum temperature T_{max}(°C) and weight loss values of the decomposition stages for 5-Bromouracil, Mg(II), Sr(II), Ca(II) and Pb(II) complexes.

Complex	Decomposition	T_{max} (°C)	Lost species	% of weight loss	
				Found	Cal
Free ligand (BrU)	one step	320	C4H2N2O2Br	99.6	100
$[Mg(BrU)_2]8H_2O$ (A)	1st step	125	$8H_2O$	25.48	26.37
	2nd step	327			
	3rd step	450			
	Total loss		$C_8H_4Br_2N_4O_3$	70.23	66.67
	Residue		MgO	6.74	7.32
$[Sr(BrU)_2(H_2O)_2]$ (B)	1st step	190	$2H_2O$	8.02	7.14
	2nd step	225			
	3rd step	337			
	Total loss		$C_8H_4Br_2N_4O_3$	71.18	72.27
	Residue		SrO	21.74	20.56
$[Ca(BrU)_2.2H_2O]H_2O$ (C)	1st step	130	H_2O	4.1	3.97
	2nd step	227	$2H_2O$	8.74	7.58
	3rd step	421			
	Total loss		$C_8H_4Br_2N_4O_3$	74.12	76.66
	Residue		CaO	12.86	11.79
$[Pb(BrU)_2]$ (D)	One step	400			
	Total loss		$C_8H_4Br_2N_4O_2$	58.16	59.26
	Residue		PbO_2	42.36	40.7

Table 6: Antibacterial and antifungal activities of 5- bromouracine (BRU) and some of its metal complexes in terms of inhibition zone diameter (mm) and % activity index.

Organism	Bacteria				Fungi			
	E.coli		*Staph.aureus*		*Asper.niger*		*Asper.ocherattious*	
	Inh.zone, mm	% Ac.Ind	Inh.zone, mm	% Ac.Ind	Inh.zone, mm	% Ac.Ind	Inh.zone, mm	% Ac.Ind
DMF	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Ampicillin	15	100	20	100	20	100	20	100
(BrU)	14.6	97.33	19.6	98	19.5	97.5	19.0	95
$[Mg(BrU)_2]$	13.7	91.33	19.4	97	19.0	95.0	19.5	97.5
$[Hg(BrU)_2]$	13.4	89.33	18.9	94.5	19.8	99.0	19.3	96.5

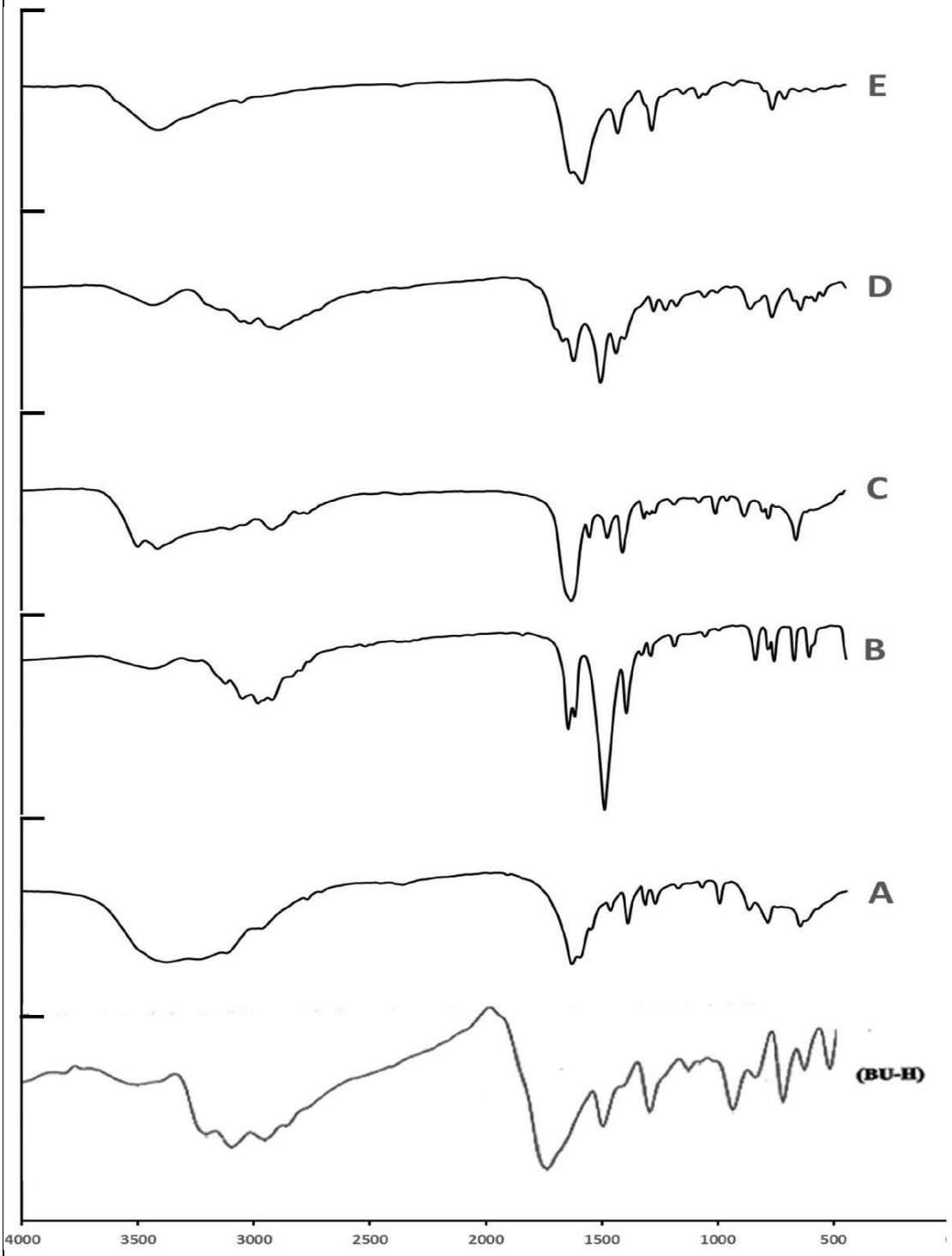


Fig. 5: IR Spectra of free ligand 5-bromouracil and complexes A, B, C, D and E.

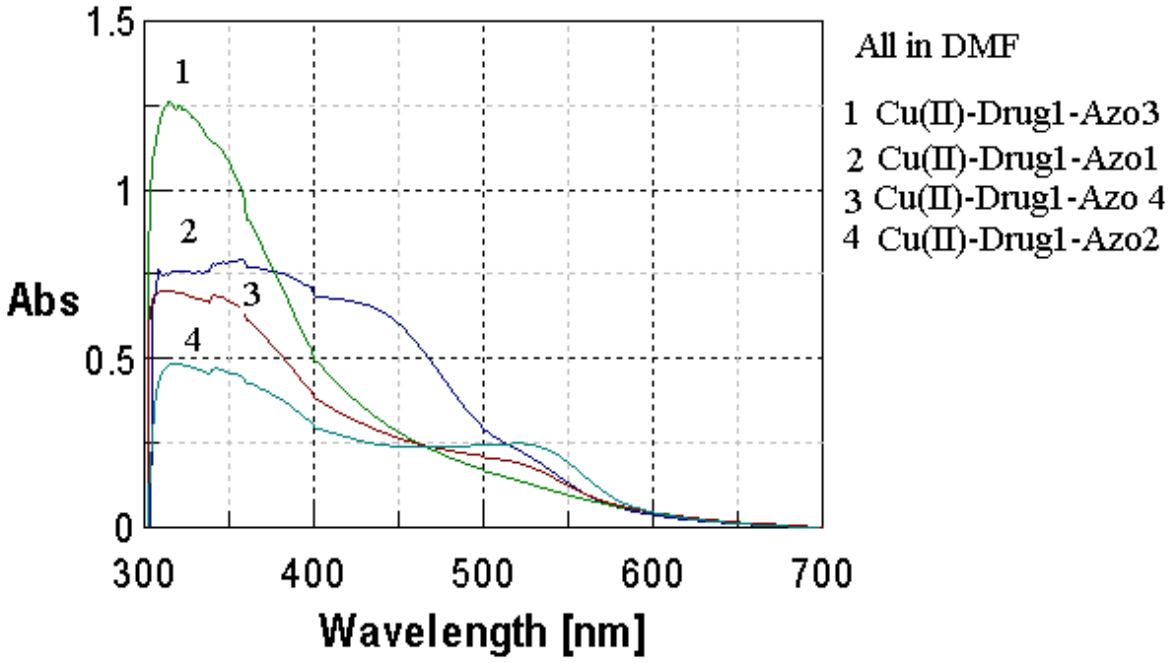
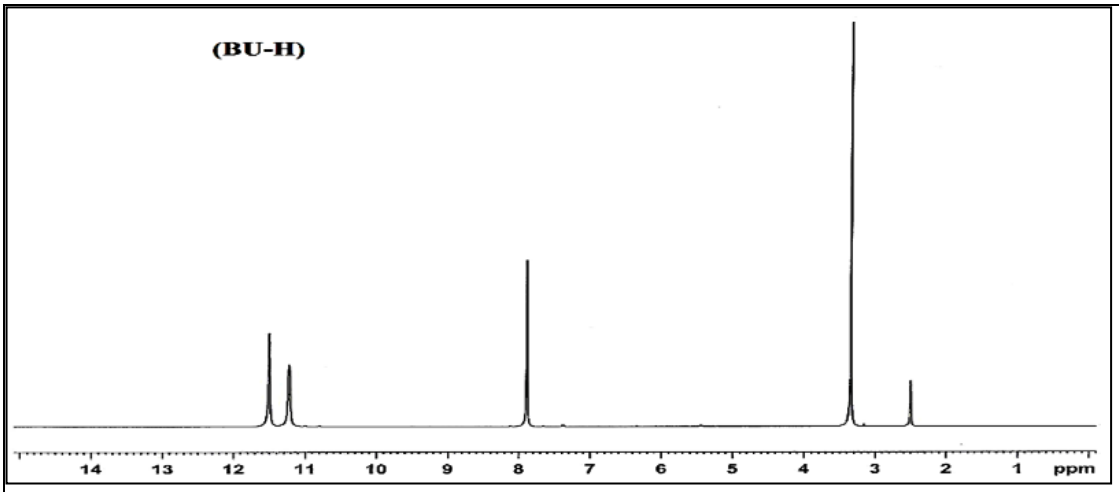
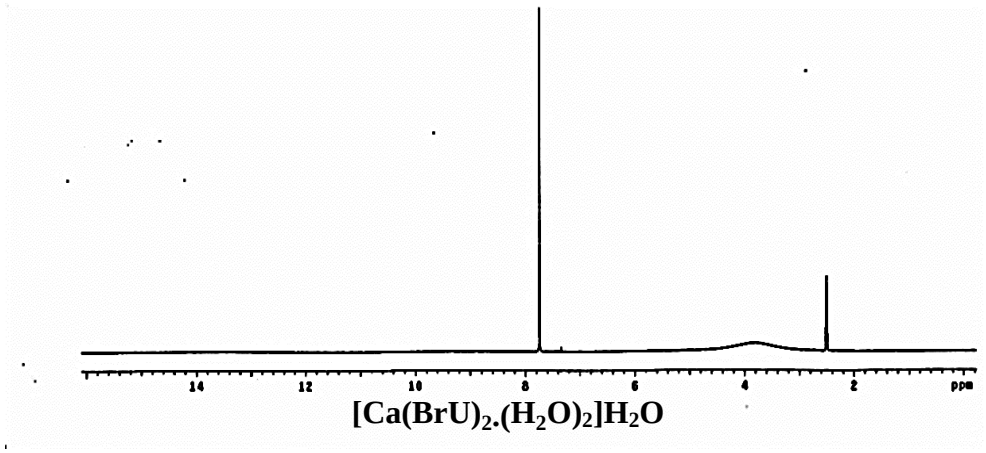


Fig.(6): Electronic spectra of 5-bromouracil (5-Br-U-H) and its Mg(II), Sr(II) and Ca(II) complexes in DMF





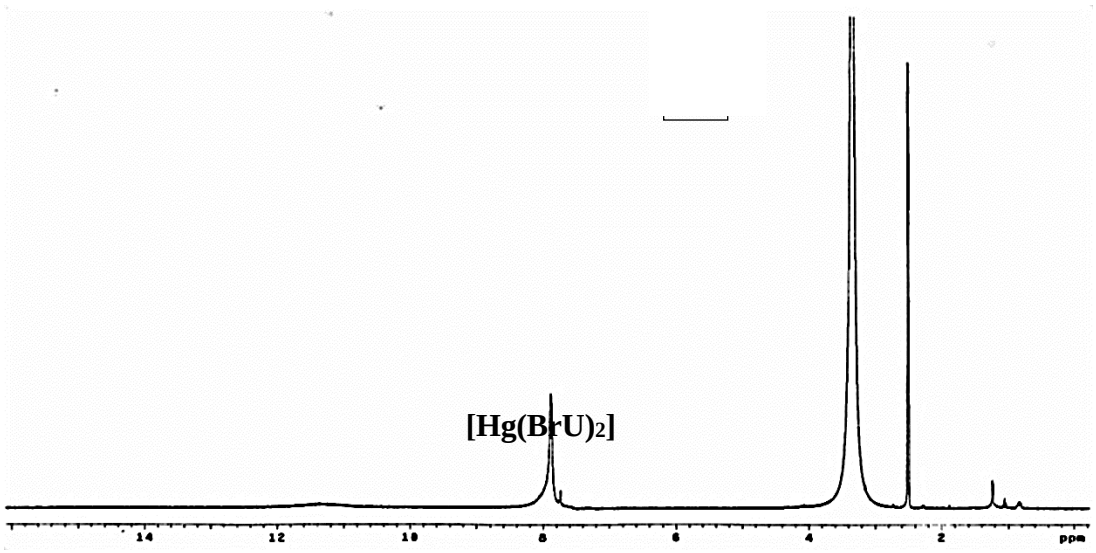
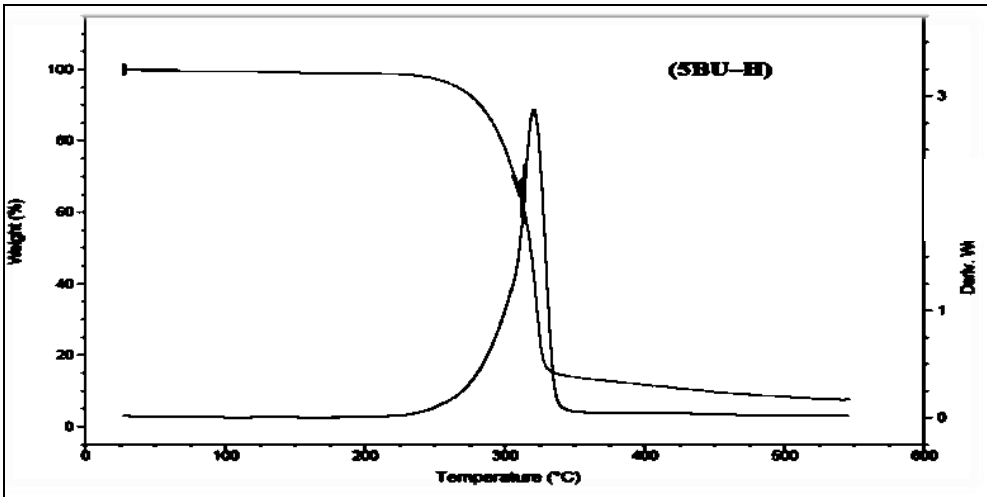
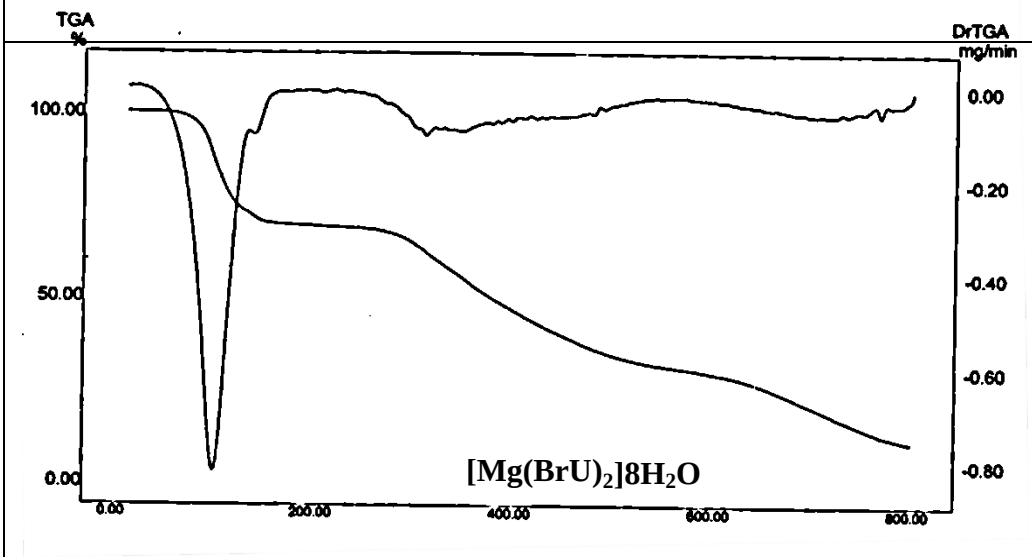


Fig. (7): *^{1}HNMR* Spectra of free ligand **5-bromouracil** and its complexes.


(5BU–H)
Weight (%)
Deriv. Wt
Temperature (°C)


TGA
%
DrTGA
mg/min
100.00
50.00
0.00
0.00
-0.10
-0.20
[Pb(BrU)2]


TGA
%
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mg/min
100.00
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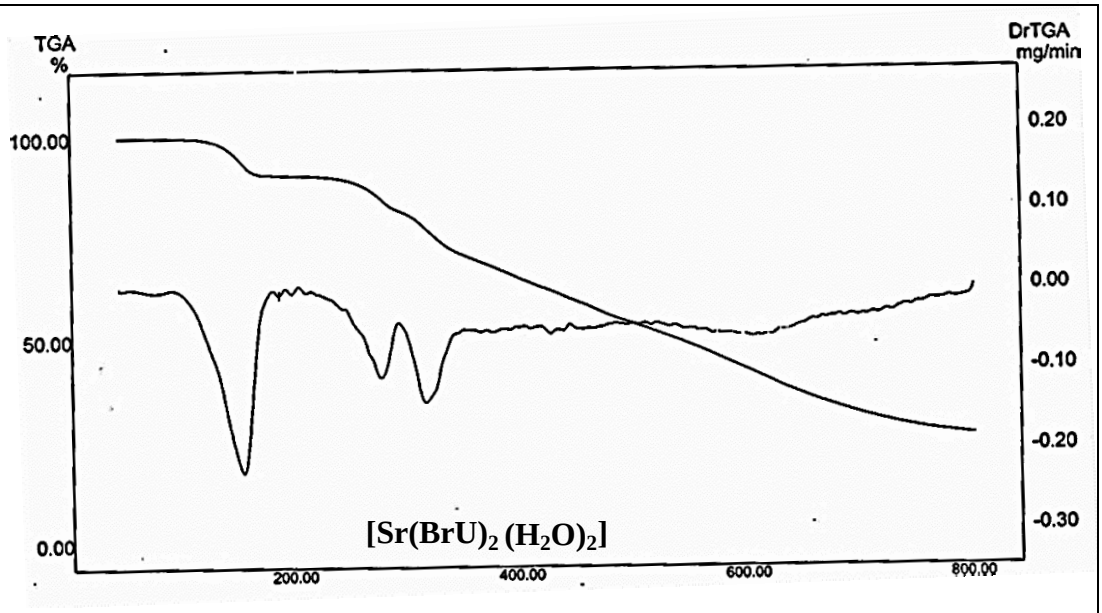
[Mg(BrU)2]8H2O



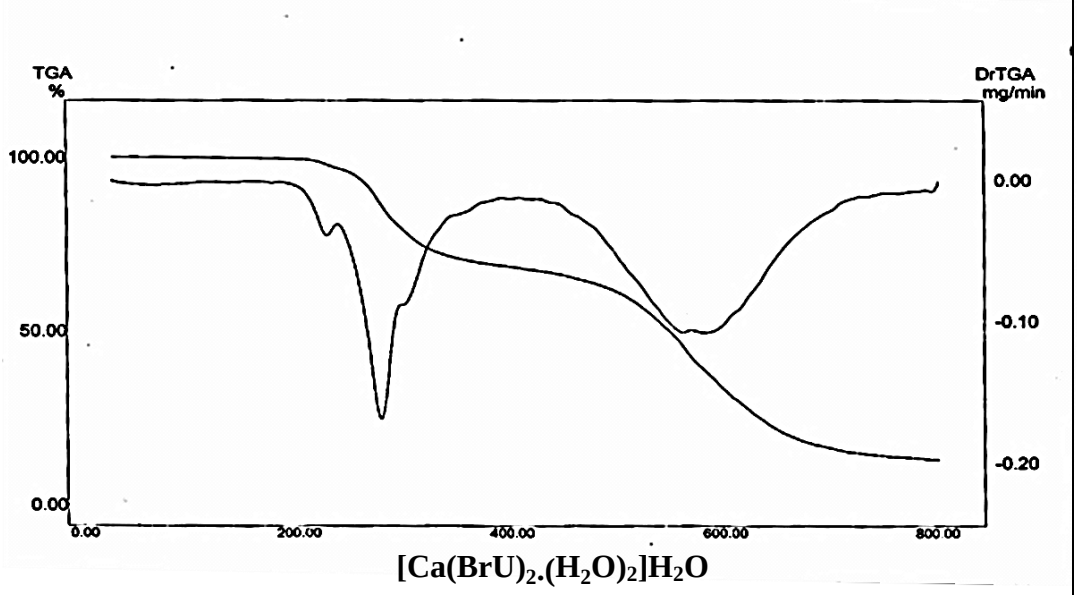


Fig (8): Thermogravimetric Analyses (TGA, DTG) of free ligand 5-bromouracil and its complexes.

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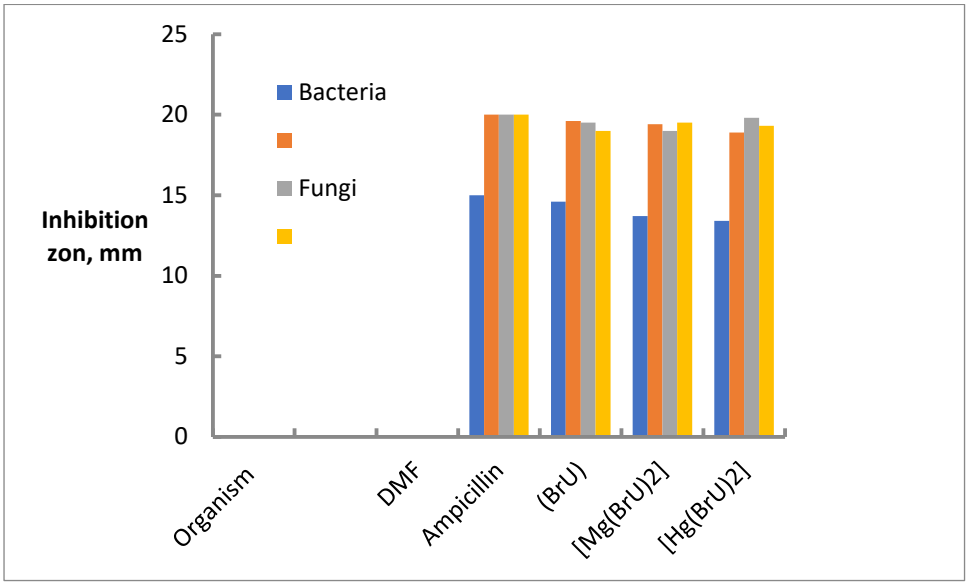

Fig. (9): Antibacterial and antifungal activities of 5- bromouracine (BRU) and some of its metal complexes in terms of inhibition zone diameter (mm).

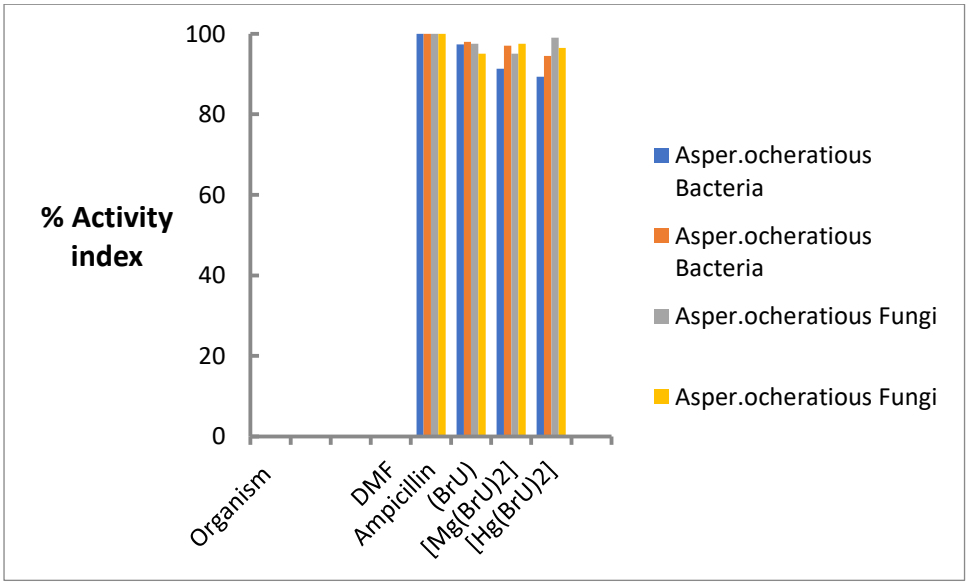

Fig. (10): Antibacterial and antifungal activities of 5- bromouracine (BRU) and some of its metal complexes in terms of % activity index.

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