



Synthesis, spectral, magnetic, Thermogravimetric and XRD studies of Oxovanadium(IV) and Dioxouranium(VI) complexes with some new Heterocyclic Schiff bases

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ABSTRACT

Oxovanadium(IV) and Dioxouranium(VI) complexes of Schiff bases derived from 3-substituted-4-animo-5-mercapto-1,2,4-triazole and glyoxal/biacetyl /benzyl have been synthesized and characterized on the basis of analytical and spectroscopic studies. The elemental analysis agrees well with the 1:1 stoichiometry of the type VO.L.So₄ and UO₂.L.(CH₃COO)₂ respectively. The IR data suggests that, ligands have coordinated to the metal ion through azomethine nitrogen atoms in a bidentate fashion by keeping sulphur atom of mercapto groups away from coordination. Thermogravimetric measurements and XRD pattern have also been done on some oxovanadium (IV) complexes. Some of the VO(IV) and UO₂(IV) complexes have been screened for their antimicrobial and antifungal activity. One of the VO(IV) complex has been evaluated for its oxytocic and antihistaminic activity.

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Introduction

Oxovanadium (IV) and Dioxouranium (VI) complexes of Schiff bases are well documented in the literature [1-8]. However, it is evident from the literature that, no attempts have been made to synthesize the complexes of Oxovanadium(IV) and Dioxouranium(VI) with Schiff bases derived from 3-substituted-4-animo-5-mercapto-1, 2, 4,-triazoles and glyoxal/ biacetyl/benzyl.

In a continuation of our investigation of the transition metal complexes of triazole Schiff bases [9, 10] we report here a study of the complexes of Oxovanadium(IV) and Dioxouranium(VI) complexes of Schiff bases derived from 3-substituted-4-animo-5-mercapto-1, 2, 4,-triazoles and glyoxal/biacetyl/benzyl. The Schiff bases and their metal complexes have been characterized by various spectroscopic and analytical studies. Some of the compounds have been screened for their antimicrobial activities.

Experimental

Materials and method:

All the chemicals used for preparing the triazoles and their Schiff bases were of reagent grade. Vanadyl sulphate monohydrate and uranyl acetate dehydrate were prepared according to the method reported earlier from our laboratory [11]

Synthesis of oxovanadium (IV) complexes

Vanadyl sulphate (0.01 mol) in the minimum amount of water was added to the Schiff base (0.01 mol) in an ethanoic medium. The reaction mixture was refluxed for an hour. Then to the reaction mixture 2mmol of sodium acetate was added and refluxion was continued for about 2 hrs and the precipitation of the complex was initiated by adding water to the reaction mixture. The precipitated complex was filtered, washed with water containing alcohol and dried under vacuum over fused calcium chloride.

Synthesis of Dioxouranium (VI) Complexes

Uranyl acetate (0.01 mol) was refluxed with 0.01 mol of Schiff base in ethanoic medium for about 3 hrs. Then, the reaction mixture was evaporated somewhat and precipitation of the complex was initiated by adding water containing ammonia drop by drop. The complex was filtered, washed thoroughly with water containing a little alcohol and dried under vacuum over fused calcium chloride.

Elemental Analysis

Vanadium and uranium content in the complexes were determined respectively by volumetry and gravimetry [12, 13]. Nitrogen was estimated by Duma's method and sulfur was determined gravimetrically as the sulphate after oxidizing with fuming nitric acid.

Physical measurements

The magnetic susceptibility of the complexes was measured at room temperature on a Gouy balance using Hg CO (SCN)₄ as calibrant. The IR spectra of the ligands and their complexes in nujol mull were recorded on a HITACHI-270 spectrophotometer in the 4000-250 cm⁻¹ region. Electronic spectra of the complexes in DMF were measured on HITACHI-model-200 spectrophotometer and a spectra of representative complexes in nujol mull were recorded on a DMF-spectrophotometer in the 350-1500 nm region. The PMR spectra of some ligands and their dioxouranium (VI) complexes in DMSO were recorded on a varian V-60 spectrometer using TMS as an internal reference. ESR spectra of oxovanadium (VI) complexes in powder form were recorded on a varian ESR spectrometer using DPPA as reference. Du part 9900 computer/thermal analyzer with 951 TG module thermobalance was used for recording TG curves. The X-ray powder diffraction data were recorded on a DE-CER P12 X-ray machine with a diffractogram.

Results and Discussion

Oxovanadium(IV) complexes are leaf green to olive green in colour, whereas, Dioxouranium(VI) complexes are lemon yellow in colour. They are not soluble in common organic solvents; however, they are soluble to a limited extent in DMF and DMSO. The elemental analysis agrees well with the 1:1 stoichiometry of the type VO.L.S_o4 and UO₂.L.(CH₃COO)₂ (Table 1). The molar conductance values in DMF are too low to account for any dissociation of the complexes. Hence, these can be regarded as non-electrolytes.

Infrared spectra

The important IR frequencies which characterize the complexes are discussed below.

The complexes exhibit broad medium bands in the region 3280-3115 cm⁻¹ followed by weak broad band around 2400 cm⁻¹ due to ν (SH) vibrations. The band due to ν (C=S) around 740 cm⁻¹ in the ligands is unaffected in the complexes indicating that, sulfur has not taken part in formation of the complexes. The strong band due to ν (C=N) appears at 1640 ± 5 cm⁻¹ of the ligands is observed in the region 1620-1610 cm⁻¹ in the complexes suggesting that, C=N group of the ligands have coordinated to the metal ion through nitrogen.

The oxovanadium(IV) complexes exhibit a strong absorption band in the region 1230-1030 cm⁻¹ and at 970 cm⁻¹ indicates the chelating bidentate nature of SO₄²⁻ in the oxovanadium(IV) complexes [14]. A high intensity band is expected for ν (V=O) vibration at 985 cm⁻¹. This reflects on high π-band order of vanadium to oxygen link of VO⁺² and indicates the presence of monomeric oxovanadium species [15, 16]. Dimerisation through (V=O) reflects in shifting of ν(V=O) results in splitting of (V=O) vibration [18]. In these oxovanadium (IV) complexes, we observe a high to medium intensity bands in the region 990-980 cm⁻¹ and these are attributed to the (V=O) vibrations.

Uranyl ion is reported to exhibit three characteristic bands at ν₁(sym) = 860 cm⁻¹, ν₃(asym)=930 cm⁻¹ and ν₂ (bend) = 210 cm⁻¹ [19]. Zones and Pennamen have reported a band in the region 940-850 cm⁻¹ for linear (O=M=O) moieties [20]. Patel [21] and Biradar et.al [22] locate ν₃ band at 920 ± 5 cm⁻¹ for acetate and Schiff base complexes of dioxouranium(VI).

Considering these assignments, we have assigned a band of high intensity in the region 930-910 cm⁻¹ to the ν₃ (asym) of O=U=O. The O=U=O bonding vibration ν₂ for these complexes is located in the region 250-210 cm⁻¹ as a medium intensity band. Monodentate coordination of the acetate in UO₂(VI) complexes of the ligands (I – XII) suggested, since ν_{as} and ν_{sym} of the carboxylate were detected, that comparable frequencies to those reported for the monodentate acetate of UO₂(VI). Bands in the region 1580- 1575 cm⁻¹ and 13315-1310 cm⁻¹ region are considered as due to the ν_{as} (COO⁻) and ν_{sym} (COO⁻) respectively.

The differences of the order of 265-260 cm⁻¹ between these frequencies is suggestive of monodentate behaviour of acetate moiety [23]. The new medium to high intensity bands appearing in the region 540-520 cm⁻¹ and 425-320 cm⁻¹ for oxovanadium(IV) complexes and in the region 525-510 cm⁻¹ for dioxouranium(VI) complexes are regarded as due to the ν_(M-N) vibrations respectively.

Thus, the IR data indicates that, the Schiff bases I–XII behave as a bidentate ligands coordinating through NN donor system in these complexes.

Magnetic data

The magnetic moments obtained for these oxovanadium(IV) complexes at room temperature are listed in Table I. The μ_{eff} values obtained for these complexes are in the range of 1.65 to 1.76 B.M. These are in the vicinity of spin only value of oxovanadium(IV) complexes hitherto reported. These results preclude the existence of exchange interactions in the complexes. The polymeric complexes with spin-spin interactions exhibit lower than normal magnetic moments and polymeric complexes with normal magnetic moments have also been reported. All the dioxouranium complexes are diamagnetic.

Electronic spectra

Oxovanadium (IV) complexes

The observed band maxima of these complexes are listed in Table II along with their ligand field and NSH (Normal Spectral Hamiltonian) parameters.

These oxovanadium (IV) complexes exhibit band in the following regions; 13110-12885, 15873 – 15503 and 24691 – 23895 cm⁻¹.

Quite diverse models have been proposed to interpret the electronic spectra of oxovanadium (IV) complexes [31-37]. The spectra of these complexes have been interpreted by using the Ballhausen and Gray (BG) model and the assignments have been made accordingly. Wasson's model cannot be used because, the difference between ν₁ and ν₂ is less than 4000 cm⁻¹. The spectra of these complexes are comparable with well studied oxovanadium (IV) complexes of coordination number five. The parameters Dq and Dt have been used to calculate the degree of dissociation of the complexes by applying the NSH theory advanced by lever et.al; [38,39]. The parameters of this theory have been fully used to relate them to those of the classical Hamiltonian theory. The parameters of both the Hamiltonians are related as follows [38]

$$DT = \frac{7\sqrt{15}dt}{2}$$

$$DQ = (6\sqrt{21} Dq - \frac{7\sqrt{21} Dt}{2})$$

$$DS = -7Ds$$

The advantage of this theory are –

- It takes into account of digital contribution to Dt.
- DQ is a measure of an average ligand field experienced by the metal ion unlike classical 10 DQ values which are measure of an in plane ligand field.
- The ratio of DQ / DT gives degree of tetragonal distortion.

The values of Ds and Dt reflect upon the σ and π-donating abilities of the ligands. The ratio DT / DQ calculated for these complexes fall in the range of 0.03058 – 0.03496. These values are lower than the values postulated for square planar complexes (viz., 0.04226) a limiting case of tetragonally distorted molecule. Hence, the complexes are moderately distorted and metal atom lies slightly above the molecular plane. The reason for this distortion may be attributed to the non-equivalences of donor atoms.

Dioxouranium (VI) complexes

Electronic spectra of the ligands in DMF exhibit intense band maxima in the region 305 – 307 nm and these involve changes of vibrational energy state in hydrogen bonded chelate ring [40] and also indicate that, thione tautomerism in the ligands.

The electronic spectra of dioxouranium (VI) complexes with these ligands display modified broad multicomponent bands having very high intensity in the region 267 – 309, 345 – 374 and 419 – 451 nm. The observed shift in the ligands and appearance of new bands in the region 345 – 374 and 419 – 451 nm in the spectra of the complexes may be contemplated for the complex formation. It has been established^[41, 42] that, the electronic spectra of dioxouranium (VI) complexes display band maxima between 340 – 500 nm region which defines the vibronic structure and determines the triatomic UO_2^{2+} moiety. In these complexes, the strong bands appearing correspond to charge transfer transitions from equatorial ligands Viz., ($\text{II} - \text{orbitals}$ of ligand to 'f' of uranium). Similar observations have been made by Vidall et.al^[43]. In the case of dioxouranium (VI) complexes of Schiff bases. The broad band maxima observed around 450 nm may be looked upon as due to the triatomic UO_2^{2+} moiety.

ESR Spectra

The ESR Spectra of these oxovanadium (IV) complexes show 8 – line spectra (^{51}V , $I = 7/2$). The 'g' and 'A' values calculated are presented in Table III and are in good agreement with the values reported for well characterized complexes^[44].

The ESR and electronic spectra have been correlated by using the expressions given by Assour et.al.,^[45]. By using 'g' values and the band maxima ν_1 and ν_2 observed for these complexes, the spin – orbit coupling coefficient (λ) has been calculated (Table III). The average λ values obtained for these complexes are in good agreement with the reported values^[46, 47]. The 'G' value has been calculated by the following equation;

$$G = g_{11} - 2.00277 / g_1 - 2.00277$$

This quantity can be used to know the order of energy levels in the electronic spectra tentatively. It has been suggested that, If $G > 4$ then, b_2 (2B_2) is above eg (2E), and normal order prevails. If $G < 4$ then, b_2 (2B_2) is below eg (2E) level. In these complexes, it is observed that the 'G' value is less than 4 and hence, it is concluded that in these complexes b_2 (2B_2) is below eg (2E). all these observations also support the electronic spectral observations.

PMR spectra

The PMR spectra of two ligands I – IX and their dioxouranium (VI) complexes have been chosen for the study.

The broad signal at 13.63 and 13.60 ppm due to NH proton of the ligands I – IX is observed at 13.65 in complexes indicating that, ligand preserve the thiol form and non-involvement of sulphur in the coordination with the metal ion. The characteristic resonance due to the azomethine proton in the complex of ligand – I appears at 8.45 ppm indicating the downfield shift with the corresponding ligand – I (8.2 ppm) this is due to deshielding. This observation renders the proof for the coordination of azomethine group to the metal ion through nitrogen. In case of complex of ligand – IX the coordination of C=N group to the metal ion through nitrogen has been inferred from the down field shift of the aromatic proton signals in the region 7.61 – 7.94 ppm with respect to the ligand – IX (6.9 – 7.9 ppm). All these observations support the IR conclusions.

Thermal studies

The thermogram of one representative complex has been studied. The thermal behavior and kinetic data obtained for this complex is listed in Table IV & Table V.

The thermogram is characterized by two horizontal lines in the ranges of the temperature 0 – 90°C and 470 – 710 °C. This amounts to the meaning that, the complex is stable up to 90°C

and after this temperature, the complex loses its weight with increase in temperature from 90 – 170°C. The loss of weight is about 5%. This can be ascribed to the melting of the complex. The cumulative weight change reaches maximum (80%). There is loss in weight of the complex by increasing the temperature from 470 – 710°C. This makes one to conclude that, the oxide form is thermally stable and does not undergo further oxidation.

Freeman and Corroll method^[48] has been used to evaluate the order of the reaction and energy of activation from a single experimental curve.

XRD studies

The X – ray powder diffraction of two representative oxovanadium (IV) complexes have been studied. The 20 values for prominent peaks have been indexed and their Q_{obsd} values have been compared with the Q values calculated ones (Table VI and VII).

The X – ray powder diffraction lines observed for oxovanadium (IV) complexes have been satisfactorily indexed on the basis of hexagonal unit lattice. The lattice parameters computed for complex No. 2 is $a = 14.3220\text{Å}$, $c = 33.1503\text{Å}$ and for complex No. 10 is $a = 11.2690\text{Å}$, $c = 35.5230\text{Å}$.

Biological activity

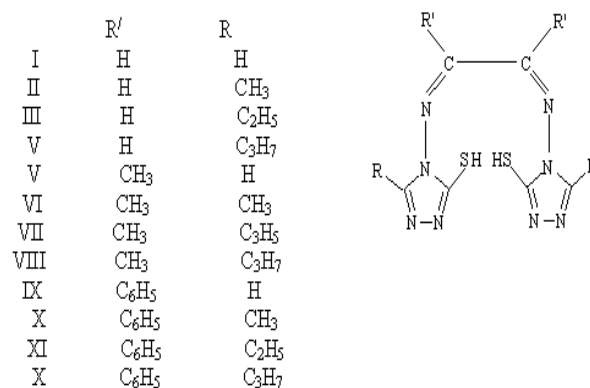
Triazoles and their derivatives have been proved to be effective bactericides^[49], pesticides^[50], fungicides^[51, 52] and insecticides^[53, 54]. 3-Amino-1,2,4-triazoles under the trade name Amizol is a commercial herbicide^[55, 56]. Antimicrobial activity of triazole derivatives and some metal complexes have been reported from our laboratory^[11, 57, 58]. They have exhibited promising results.

With this background of information, we have screened some of the oxovanadium (IV) and dioxouranium (VI) complexes for their antimicrobial and pharmacological activities (Table VIII and Table IX).

Antimicrobial activity

Some of the oxovanadium (IV) and dioxouranium (VI) complexes have been assessed for their antibacterial activity against E. coli and B. cirrafigellosus (Bacteria and antifungal activity against C. albicans and A. niger). The data in the table VII indicates that, the antimicrobial activity of most of the oxovanadium (VI) complexes is more than that of the free ligands and dioxouranium (VI) complexes.

Figure 1. Structure of Schiff bases



Pharmacological activity

One representative complex of oxovanadium (IV) of ligand – II has been evaluated for its oxytocic activity and anti – histaminic activity.

From the Table – VIII it is clear that, oxovanadium (IV) complex showed no inhibition of oxytocic activity on isolated

rat uterus, whereas, it has got 22.85% of antihistaminic activity on guinea pigilum.

Analytical data and physico chemical studies suggest the following structures for these complexes in which oxovanadium (IV) is pentacoordinated and dioxouranium (VI) hexacoordinated.

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Table I. Elemental Analysis, Magnetic moment and molar concentration Data of Oxovanadium (IV) and Dioxouranium (VI) complexes of 3-substituted-4-amino-5-mercapto-1,2,4-trizole Schiff bases.

-	Empirical formula	M%		N%		S%		Magnetic Moments μ_{eff} B.M	Molar conductance data $\text{Ohm}^{-1} \text{cm}^2 \text{mole}^{-1}$
		Calcd.	Obtd.	Calcd.	Obtd.	Calcd.	Obtd.		
1	(C ₆ H ₆ N ₈ S ₂)VO.SO ₄	12.21	12.22	26.86	26.88	23.02	23.05	1.68	12.98
2	(C ₈ H ₁₀ N ₈ S ₂)VO.SO ₄	11.45	11.46	25.17	25.19	21.57	21.58	1.70	16.27
3	(C ₁₀ H ₁₄ N ₈ S ₂)VO.SO ₄	10.77	10.75	23.68	23.63	20.29	20.30	1.69	14.81
4	(C ₁₂ H ₁₈ N ₈ S ₂)VO.SO ₄	10.17	10.15	22.35	22.32	19.16	19.20	1.65	11.36
5	(C ₈ H ₁₀ N ₈ S ₂)VO.SO ₄	11.45	11.48	25.17	25.18	21.57	21.59	1.66	17.49
6	(C ₁₀ H ₁₄ N ₈ S ₂)VO.SO ₄	10.77	10.78	23.68	23.70	20.29	20.31	1.74	16.00
7	(C ₁₂ H ₁₈ N ₈ S ₂)VO.SO ₄	10.17	10.13	22.35	22.33	19.16	19.13	1.72	16.00
8	(C ₁₄ H ₂₂ N ₈ S ₂)VO.SO ₄	9.63	9.60	21.17	21.18	18.14	18.15	1.75	13.36
9	(C ₁₈ H ₁₄ N ₈ S ₂)VO.SO ₄	8.95	8.97	19.68	19.65	16.87	16.85	1.70	16.73
10	(C ₂₀ H ₁₈ N ₈ S ₂)VO.SO ₄	8.53	8.50	18.76	18.75	16.08	16.04	1.68	15.38
11	(C ₂₂ H ₂₂ N ₈ S ₂)VO.SO ₄	8.15	8.17	17.92	17.93	15.36	15.35	1.76	10.57
12	(C ₂₄ H ₂₆ N ₈ S ₂)VO.SO ₄	7.80	7.81	17.15	17.18	14.70	14.68	1.65	13.40
13	(C ₆ H ₆ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	37.07	37.20	17.45	17.51	9.97	9.92	-	18.77
14	(C ₈ H ₁₀ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	35.52	35.81	16.72	16.82	9.55	9.51	-	18.41
15	(C ₁₀ H ₁₄ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	34.09	34.15	16.05	16.18	9.17	9.20	-	18.90
16	(C ₁₂ H ₁₈ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	32.78	32.91	15.43	15.51	8.82	8.83	-	17.34
17	(C ₈ H ₁₀ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	35.52	35.71	16.72	16.74	9.55	9.52	-	14.01
18	(C ₁₀ H ₁₄ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	34.10	34.22	16.05	16.10	9.17	9.20	-	14.20
19	(C ₁₂ H ₁₈ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	32.82	32.82	15.43	15.52	8.82	8.84	-	14.05
20	(C ₁₄ H ₂₂ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	31.56	31.65	14.85	14.91	8.49	8.51	-	16.90
21	(C ₁₈ H ₁₄ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	29.97	30.02	14.11	14.15	8.06	8.09	-	13.38
22	(C ₂₀ H ₁₈ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	28.95	29.02	13.63	13.71	7.79	7.75	-	16.31
23	(C ₂₂ H ₂₂ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	28.00	28.02	13.18	13.22	7.52	7.54	-	16.61
24	(C ₂₄ H ₂₆ N ₈ S ₂)UO ₂ .(CH ₃ COO) ₂	27.10	27.25	12.76	12.82	7.29	7.31	-	14.53

TABLE II. Electronic spectral data (in cm^{-1}) of oxovanadium (IV) complexes of 3-substituted-4-amino-5-mercapto-1,2,4-triazole Schiff bases.

C. No	v			Dq	Ds	Dt	DQ	DS	DT	DT/DQ
1	130222	15503	24038	1550.3	-3079.57	756.65	30490.17	21557	10256.82	0.3363
2	13075	15508	24685	1550.8	-3178.85	707.68	31289.37	22252	9592.01	0.3065
3	13071	15503	24691	1550.3	-3179.85	706.28	31298.08	22259	9574.01	0.3058
4	13095	15515	23895	1551.5	-3067.85	778.28	30176.26	21475	10550.01	0.3496
5	13023	15503	24038	1550.3	-3079.57	756.65	30490.17	21557	10256.82	0.3364
6	13110	15510	24060	1551.0	-3094.28	765.42	30368.73	21660	10375.72	0.3416
7	13089	15873	24330	1587.3	-3078.00	771.00	31277.46	21546	10451.24	0.3341
8	13035	15510	24045	1551.0	-3081.42	758.14	30485.59	21570	10276.96	0.3371
9	12987	15503	24271	1550.3	-3107.85	732.68	30874.6	21755	9931.87	0.3216
10	12885	15510	24275	1551.0	-3092.35	721.28	31076.74	21650	9777.34	0.3146
11	13055	15535	24295	1553.5	-3116.42	741.14	30826.99	21815	10046.52	0.3259
12	12895	15535	24310	1553.5	-3095.71	721.57	31140.89	21670	9781.21	0.3140

Table III. ESR parameters of oxovanadium (IV) complexes of 3-substituted-4-amino-5-mercapto-1,2,4-triazole Schiff bases.

Complex No.	g_a	g_{11}		A_{avG}			G	λ
2	1.940	1.922	1.977	86.55	157.92	50.87	3.19	162.30
10	1.939	1.919	1.979	86.81	158.56	50.94	3.52	157.20

Table IV. Thermogravimetric data for oxovanadium (IV) complexes of 3-substituted-4-amino-5-mercapto-1,2,4-triazole Schiff Base.

Complex No	Empirical Formula	Decomposition Temp in $^{\circ}\text{C}$	% weight Obs.	Loss Expected	Inference	Order reaction	Energy Activation
2	$(\text{C}_8\text{H}_{10}\text{N}_8\text{S}_2)\text{VO.SO}_4$	180	5.60	5.86	Loss of Glyoxal	1.45	23.38
		440	56.25	57.79	Loss of Triazole moiety		
		470	62.35	63.21	Loss of Ligand Molecule		

Table V. Kinetics of Thermal Decomposition Studies Of oxovanadium (IV) complexes of 3-substituted-4-amino-5-mercapto-1,2,4-triazole Schiff base

Complex No	Empirical formula		
2	$(\text{C}_8\text{H}_{10}\text{N}_8\text{S}_2)\text{VO.SO}_4$	1.62490000000000	1.54500000000000
		1.0880	-6.1600
		1.2116	2.4187
		1.2220	1.7540
		2.7840	18.7900
		0.8790	-6.3620

Table VI. Observed and calculated Q and h k l values of Oxovanadium (IV) of 3-substituted-4-amino-5-mercapto-1,2,4-triazole Schiff bases

Sl. No	2 θ	d	RI*	Q obs	Q calcd	h k l
1	13.458	6.579	145	0.0231	0.0231	1 1 2
2	19.050	4.688	195	0.0455	0.0462	1 2 0
3	22.713	3.930	129	0.0777	0.0654	1 0 8
4	24.788	3.587	64	0.0647	0.0776	1 1 8
5	27.159	3.285	147	0.0927	0.0929	3 1 3
6	28.066	3.177	190	0.0991	0.0991	3 1 4
7	29.030	3.087	148	0.1049	0.1059	4 0 1
8	32.073	2.783	392	0.1291	0.1286	3 1 7
9	33.906	2.647	257	0.1427	0.1433	3 1 8
10	38.542	2.333	107	0.1837	0.1836	3 3 3
11	48.736	1.869	151	0.2863	0.2869	5 2 6
12	50.652	1.803	78	0.3077	0.3084	6 0 9

RI* = Relative intensity

System hexagonal; a = 14.3220Å c = 33.1503Å

Cell volume = 5888.6087

Table VII. Observed and calculated Q and h k l values of Oxovanadium (IV) complexes of 3-substituted-4-amino-5-mercapto-1,2,4-triazole Schiff bases

Sl. No	2θ	d	RI*	Q obs	Q calcd	h k l
1	9.952	8.839	924	0.0128	0.0127	0 0 4
2	13.449	6.551	783	0.023	0.0231	1 0 4
3	15.932	5.564	554	0.0323	0.0324	1 1 1
4	22.536	3.928	368	0.0648	0.0645	0 0 9
5	27.417	3.253	458	0.0945	0.0947	3 0 0
6	28.976	2.955	364	0.1145	0.1148	2 0 9
7	30.295	2.817	445	0.1260	0.1271	3 0 5
8	31.887	2.620	512	0.1457	0.1469	2 2 0
9	34.337	2.617	358	0.1460	0.1469	2 2 5
10	37.862	2.376	251	0.1772	0.1774	2 2 8

RI* = Relative intensity

System hexagonal; a = 11.2690Å c = 35.5230Å

Cell volume = 3906.594

Table VIII. Results of antibacterial and antifungal activity of oxovanadium (IV) and Dioxouranium (VI) complexes

Ligand number	Complex number	Activity against (Zone of inhibition in mm)			
		Bacteria		Fungi	
		E.coli	B.cirrafigellosus	C.albicans	A.niger
Oxovanadium (IV) complexes					
I	1	12	18	17	20
II	2	18	16	16	13
III	3	20	16	18	13
V	5	17	15	15	20
VI	6	14	14	13	18
VII	7	13	12	17	20
VIII	8	15	14	16	19
Dioxouranium (VI) complexes					
I	1	13	10	16	13
II	2	17	16	20	13
III	3	19	18	21	17
V	5	17	15	15	21
VI	6	15	10	15	13
VII	7	15	15	16	18
VIII	8	14	16	17	15
(DMF 0.1 ml)		11	11	11	11
Solvent control					
Sulphamethoxazole (20 mcg)		20	20	-	-
Gentamycin (10 mcg)		24	24	-	-
Nystalin (50 IU)		-	-	23	25

Table IX. Data showing the Oxytocic Activity and Antihistaminic Activity of Oxovanadium (IV) Complexes

Sl. No	Volume of test solution and Oxytocic /Histamine added (ml)	Peak Height	Oxytocic Activity (%)	Histaminic Activity (%)
1	(C ₈ H ₁₀ N ₈ S ₂)VO.SO ₄ + 0.3 ml Std.Oxytocic	3.5	100 (0.0% inhibition)	-
2	(C ₈ H ₁₀ N ₈ S ₂)VO.SO ₄ + 0.4 ml Std.Histamine	2.7	-	77.15 (22.85% inhibition)