Coordination Chemistry of Aluminium (III) and Gallium (III) Complexes with Biaoctive Schiff Bases: Synthetic, Spectral and Biological Aspects



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Abstract Biological important complexes of aluminium (III) and gallium (III) derived from 2-hydroxy-N-phenyl benzamide hydrazine carboxamide(HO^NOH), 2-hydroxy-N-phenyl benzamide hydrazine (HO^NSH) and 2-hydroxy-N-phenyl carbothioamide benzamide hydrazine carbodithioic acid (HO^NSH) have been prepared and investigated using a combination of microanalytical analysis, melting point, electronic, IR, ¹H NMR and ¹³C NMR spectral studies, cyclic voltammetry and X-ray powder diffraction studies. Aluminium and gallium isopropoxides interact with the ligands in 1:1 and 2:3 molar ratios (metal: ligand) resulting in the formation of coloured products. On the basis of conductance and spectral evidences, a pentacoordinated structure for aluminium (III) and gallium (III) complexes have been assigned. The ligands are coordinated to the aluminium (III) and gallium (III) via the azomethine nitrogen atom and the thiolic sulfur atom/enolic oxygen atom. On the basis of X-ray powder diffraction study one of the representative gallium complex was found to have Orthorhombic Lattice, having Lattice Parameters: a= 8.9999A°, b= 14.5000A°, c= 6.9800A°. The free ligands and their metal complexes have been tested in vitro against a number of pathogenic microorganisms in order to assess their antimicrobial and pesticidal properties. Both the ligands and their complexes were found to possess appreciable fungicidal, bactericidal and pesticidal properties.

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Keywords:

Schiff bases,X-ray powder diffraction studies, thiosemicarbazone, hydrazine carbodithioic acid,2-hydroxy-N-phenylbenzamide,cyclic voltammetry,pesticidal and antimicrobial activity.

Introduction

The coordination chemistry of group 13 metal ions is of biochemical interest because of the potential use in the treatment and diagnosis of disease ¹⁻⁴. The Schiff base ligands have played an important role in the development of coordination chemistry⁵. Schiff bases are important class of ligands due to their synthetic flexibility, their selectivity and sensitivity towards the central metal atom, structural similarities with natural biological substances and also due to the presence of the imine group (N=C<) which imparts in elucidating the mechanism of transformation and rasemination reaction in biological system⁶. It is well known that several Schiff base complexes have anti-inflammatory, anti-fungal, antibacterial antiviral, and anti HIV activity ⁷⁻¹³.

Aluminum is the third abundant element in the earth inferior to oxygen and silicon, and it is widely used as building materials, water purification, food additives and clinical drug ¹⁴. In Schiff base complexes of aluminium, the coordination environment at the metal center can be modified by attaching different substituents to the ligand, providing useful steric and electronic properties essential for the fine tuning of structures and reactivity ¹⁵. Aluminium, a well known and commonly exposed neurotoxin, was found to alter glutamate and c-aminobutyrate levels as well as activities of the associated enzymes with regional specificity^{16,17}. Aluminium (III) also inhibits glutamate dehydrogenase (GDH), a central enzyme in glutamate metabolism ¹⁸. Gallium plays an important role in pharmaceuticals ¹⁹ and as antitumor ²⁰, antiviral²¹ and anticoagulant agents and thallium as a probe for K⁺ in biological systems ²². The trivalent gallium cation is capable of inhibiting tumor growth, mainly because of its

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resemblance to ferric ion ²³. Gallium (III) complexes of an aminophenol ligand are active against chloroquine and *Plasmodium falsiparum* strains²⁴.

Aim of the Study

The main aim of synthesis to correlate the structural features of the newly synthesized compounds with the effect on biochemical functions.

Second aim of the biocidal activity is to facilitate the study of microbiology. The objective of biocidal screening is to design drugs with a broader spectrum of activity particularly in clinical areas and to have low toxicity but better or equal activity as compared to the standards.

Experimental

All the chemicals used in the synthesis of the complexes were of A.R. grade. All the solvents were dried and distilled before use. Aluminium 25 and gallium 26 isopropoxides were prepared by literature methods. The ligands (L¹H₂), and (L²H₂) were prepared by the reported method 27 .

Preparation of Aluminium (III) and Gallium (III) Complexes

Aluminium (III) and gallium (III) isopropoxide and ligands were dissolved in dry benzene in 1:1 and 2:3 molar ratios. The resulting mixture was refluxed for 16-20 hours. The progress of the reaction was checked by measuring the amount of isopropanol in the azeotrope. After completion of the reaction the excess of the solvent was removed under reduced pressure and dried in vacuo. The physical properties and analytical data of these complexes are enlisted in Table 1.

Analytical Methods and Physical Measurements

The molecular weights were determined by the Rast camphor method. IR spectra of the ligands and their metal complexes were recorded with the help of Nicolet Megna FT IR 550 spectrophotometer using KBr pellets. The purity of these ligands and their metal complexes was checked by the TLC on silica Gel-G using anhydrous dimethylsulphoxide and benzene (1:1) as solvent. Isopropanol in the azeotrope and isopropoxy in the complexes were estimated by an oxidimetric method.²⁸ ¹H NMR and ¹³C NMR spectra were

¹H NMR and ¹³C NMR spectra were recorded in deuterated dimethylsulphoxide (DMSOd₆) using tetramethylsilane (TMS) as standard on a JEOL AL 300 FT NMR spectrometer. Electronic spectra of the complexes were recorded in DMF on a UV-160 A Shimadzu spectrophotometer in the range 200-600 nm. X-Ray powder diffractogram of one of the representative compounds was obtained on a Philip Model PW 1840 automatic diffractogram using Cu(Ka) target with Mg filter. The wavelength used was 1.540598 Å. Voltammetric measurements were performed with a Metrohm Computrace Voltammetric

RNI No.UPBIL/2016/67980Vol-I* Issue-VI*September - 2016complexes of
t chloroquineAnalyzer μ AUTOLAB TYPE III Potentiostat
Ecochemie (Utrecht, The Netherlands) Model 757 VA.
A conventional three-electrode system was used
consisting of an Ag/AgCl/KCI reference electrode, a
hanging mercury drop electrode (HMDE) as a working
electrode and a graphite rod as auxillary electrode.

The whole measurements were automated and controlled through the programming capacity of the apparatus. The data were treated through a PC connected to the Electrochemical Analyzer version-757 VA computrace. Nitrogen and sulfur were estimated by the Kjeldahl's and Messengers methods, respectively²⁹.

Microbial Assay Antifungal Activity

The antifungal activity was evaluated against Macrophomina phaseolina and Fusarium oxysporum using Czapek's agar medium having the composition, glucose 20g, starch 20g, agar-agar 20g and distil water 1000mL.To this medium was added requisite amount of the compounds after being dissolved in dimethylformamide so as to get a certain concentration. The medium then was poured into petri plates and the spores of fungi were placed on the medium with the help of inoculum's needle. These petri plates were wrapped in polythene bags containing a few drops of alcohol and were placed in an incubator at $30 \pm 2^{\circ}$ C. The controls were also run and three replicates were used in each case. The linear growth of the fungus was recorded by measuring the diameter of the fungal colony after 96h and the percentage inhibition was calculated by the equation:

% Inhibition = (C-T) 100 / C

Where C and T are the diameters of the fungal colony in the control and the test plates, respectively.

Antibacterial Activity

Antibacterial activity was tested against *Staphylococcus aureus* and *Escherichia coli* using the paper disc plate method. The nutrient agar medium (peptone, beef extract, NaCl and agar-agar) and 5 mm diameter paper discs of Whatman filter paper No.1 were used. The compounds were dissolved in methanol for obtaining the concentration of 500 and 1000 ppm. The filter paperdiscs were soaked in these solutions, dried and then placed in the petri plates previously seeded with the test organisms. The plates were incubated for 24 h at $28 \pm 2^{\circ}$ C and inhibition zone around each disc was measured.

Results and Discussion

The reactions that led to formation of the metal (III) Schiff-base complexes and their adducts can be represented by equations 1 and 2.

$$2 \text{ M } (\text{OPr}^{i})_{3}+2 \text{ HO}^{N}\text{N}\text{XH} \xrightarrow{\text{Dry Benzene}} \{ \text{ M } (\text{OPr}^{i})_{2}(\text{O}^{N}\text{N}\text{X}) \}_{2}+4 \text{Pr}^{i}\text{OH}$$
(1)
$$2 \text{ M } (\text{OPr}^{i})_{3}+3 \text{ HO}^{N}\text{N}\text{XH} \xrightarrow{\text{2:3}} \{ \text{ M}_{2}(\text{O}^{N}\text{N}\text{X})_{3} \}+6 \text{Pr}^{i}\text{OH}$$
(2)

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(Where, M=AI or Ga and O^NX represents the Donor set of the Ligand Molecules and X = O or S).

The resulting products are coloured solids and soluble in DMF, DMSO, THF and methanol. The molecular weight determinations by the Rast camphor method revealed the dimeric nature of metal derivatives.

Electronic Spectra

The electronic spectra of the ligands $L^{1}H_{2}$ and L²H₂ exhibit two bands at 237 and 272 nm. These bands are assignable to π - π * transitions of the azomethine group. An additional band arising from >C=N chromophore at 370 nm shift to a lower wave length for metal complexes due to coordination of the azomethine nitrogen to the metal atom ³⁰.

Infrared Spectra

The IR spectra of the free ligands and their complexes were scanned in the form of KBr pellets.

IR spectra of the free ligands show a medium intensity band at 3260-3128 cm⁻¹ due to UNH /u OH vibrations, which remain absent in the spectra of the complexes. The bands due to u(C=O) and u(C=S) modes in the spectra of the ligands are observed at 1705 $\rm cm^{-1}$ and 1035 $\rm cm^{-1},$ respectively. First two bands disappeared in the spectra of the metal compounds, suggesting the enolization and thioenolization of the ligands and their chelation through the amido oxygen and thiolic sulfur, respectively. In the ligands $L^{1}H_{2}$ and $L^{2}H_{2}$ the most significant band in the region 1620 cm⁻¹ assignable to $v(C=N)^{31}$ group shifts to the higher wave number in the complexes suggesting the coordination of the azomethine nitrogen to the metal atom. There are no changes in the u sym and u asym modes of the NH₂ group appearing at ca 3360 and 3480 cm respectively, indicating the noninvolvement of this amino group in chelation. The bands at 960-925 cm⁻¹ in the spectra of 1:1 complexes of bifunctional tridentate ligands are assignable to v C-O vibrations of bridging isopropoxy groups. The complexes exhibit new bands in the region 760-612 cm⁻¹, 590-460 cm⁻¹ and 430-300 cm⁻¹ which may be attributed to the different vibrational modes of $AI - O^{32}$, $AI \leftarrow N^{33}$ and AI-S³⁴, respectively. The gallium complexes exhibit new bands in the region 660-600 cm⁻¹,480-350 cm⁻¹ and 320–280 cm⁻¹ which may be attributed to the different vibrational modes of Ga– O, Ga \leftarrow N³⁵ and Ga-S³⁶, respectively.

¹H NMR Spectra

The ¹H NMR spectra of the ligands and their complexes have been recorded in DMSO-d6- using TMS as an internal standard. In the ligands, the signals in the region δ 12.10-12.14 ppm are due to – OH which disappear in the complexes and confirms the deprotonation and complexation. The signal due to the -NH proton attached to the phenyl ring remains unaltered in the complexes.

The NH₂ group gives singlet at δ 2.80-3.04 ppm in the ligands ($L^{1}H_{2}$ and $L^{2}H_{2}$) and their complexes.

This shows that the NH₂ group is not taking part in the complexation. The signal of -NH proton in the ligands in the range δ 10.72-10.86 ppm disappears in the spectra of the corresponding complexes. The free ligands show multiplets in the region δ 6.67-8.36 ppm attributable to aromatic protons. Chemical shift values of all the complexes are listed in Table 2.

The spectra of $\{AI(OPr^{i})(L^{1})\}_{2}$. and {AI $(OPr^{i})(L^{2})_{2}$ display doublets which are attributed to gem-dimethyl protons of the bridging isopropoxy groups. One set of signals appeared as a doublet (OCH (CH₃)₂) and septet (OCH(CH₃)₂) at δ 1.16–1.38 and 4.08-4.38 ppm, respectively, in the spectra of 1:1 complexes indicating the presence of one isopropoxy group. These signals are absent in the spectra of 2:3 complexes. ¹³C NMR Spectra

The ¹³C NMR spectral data also support the authenticity of the proposed structures. The considerable shifts in the positions of carbon atoms adjacent to the azomethine nitrogen (δ 167.15-170.06 ppm) and thiolic sulfur/enolic oxygen (δ 176.24-182.68 ppm) support the proposed coordination in the complexes. Thus the shifts in the position of carbon atoms adjacent to the coordinating atoms clearly suggest the bonding of the azomethine nitrogen and amido oxygen to the aluminium and gallium atoms.

signals for $(OCH(CH_3)_2)$ The and (OCH(CH₃)₂) carbons of isopropoxy group in the spectra of 1:1metal complexes have been observed in the region δ 73.16–75.15 and 23.24–24.92 ppm, respectively, and are not observed in the spectra of 2:3 complexes. Chemical shift values of all the complexes are listed in Table 3.

⁷AI NMR Spectra

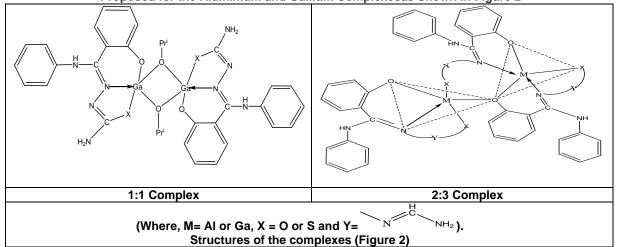
²⁷AI NMR spectra of these compounds have been recorded in $CDCI_3$ solution with reference to $AI(NO_3)_3$. The ²⁷AI NMR spectra of metal complexes have a sharp signal at δ 11.97–14.90 ppm assigned to five-coordinated aluminium ³⁷ complexes.

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D: 2455-0817 RNI No.UPBIL/2016/67980 Vol-I* Issue-VI*September - 2016 Thus, on the Basis of the Above Discussion, The Pentacoordinated Structures Can be Proposed for the Aluminium and Gallium Complexesas Shown in Figure 2



Conclusion

The ligands L^1H_2 and L^2H_2 coordinate in 1: 1, and 2: 3 metal: ligand ratio as bifunctional tridentate. Antimicrobial activity of the complexes and the ligands showed that the metal complexes are more active than the parent ligands. The fungicidal activity was better when compared to the bactericidal activity. It is interesting to note that the synthesized metal complexes with L^2H_2 have more activity than the L^1H_2 . This may be due to the presence of the sulfur atoms in such ligands. Pesticidal activity of the complexes and the ligands showed that the metal complexes are more active than the parent ligands and gallium complexes.

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Table 1

Synthetic and Analytical Data of Aluminium (III) and Gallium (III) Complexes With Bifunctional Tridentate O^N^O And O^N^S Donor Ligands.

S.	Compound	Yield	Product	M.P. (°C)	ļ	Analyses (%) Found/ (Calcd)				Mol. Wt. Found
No.		%	and Colour							/ (Calcd.)
					С	Н	Ν	S	М	
1.	${AI(OPr^{1})_{2}(L^{1})}_{2}$	82	White	250-252	57.88	6.20	13.46	-	6.42	832.56
					(57.96)	(6.32)	(13.58)		(6.51)	(828.83)
2.	{ Al ₂ (L ¹) ₃ }	79	White	268-270	58.62	4.12	19.46	-	6.14	864.52
					(58.74)	(4.23)	(19.57)		(6.29)	(858.83)
3.	${Ga(OPr^{1})_{2}(L^{1})}_{2}$	84	Light gray	110-112	51.34	4.72	14.04	-	17.42	801.36
					(51.43)	(4.83)	(14.12)		(17.58)	(794.14)
4.	{ Ga ₂ (L ¹) ₃ }	83	Gray	100-102	53.32	3.74	17.76	-	14.65	956.12
					(53.43)	(3.85)	(17.81)		(14.77)	(944.24)
5.	${AI(OPr')_2(L^2)}_2$	84	Off white	198-200	55.72	5.22	12.94	7.32	5.12	868.52
					(55.80)	(5.32)	(13.02)	(7.45)	(5.27)	(860.98)
6.	{ Al ₂ (L ²) ₃ }	82	Off white	278-280	55.54	3.92	18.42	10.52	5.82	918.12
					(55.62)	(4.00)	(18.53)	(10.61)	(5.95)	(906.97)
7.	${Ga(OPr')_2(L^2)}_2$	86	Cream	220-222	49.32	4.52	13.42	7.67	16.76	832.48
					(49.43)	(4.64)	(13.56)	(7.77)	(16.88)	(826.29)
8.	{ Ga ₂ (L ²) ₃ }	83	Cream	214-216	50.72	3.56	16.80	9.58	13.94	999.82
					(50.83)	(3.66)	(16.94)	(9.69)	(14.05)	(992.46)

Table 2

¹H NMR Spectral Data (δ Ppm) of the Ligands and their Aluminium (III) and Gallium (III) Complexes

Compound		-NH (bs)	-NH ₂ (s)	-S-CH₂	φ-NH	Aromatic	Isopropoxy groups		
	-OH (s)			(s)	(s)	protons (m)	Gem-dimethyl (d)	Methine (septet)	
L^1H_2	12.14	10.86	2.92	-	10.60	6.68 - 8.36	-	-	
L^2H_2	12.12	10.84	2.80	-	10.64	6.75 - 8.32	-	-	
${AI(OPr^{i}) (L^{1})}_{2}$	-	I	2.94	-	10.62	6.70 - 8.35	1.16(bridging)	4.38(bridging)	
${AI_2(L^1)_3}$	-	I	2.96	-	10.64	6.72 - 8.38	-	-	
{Ga(OPr ⁱ) (L ¹)} ₂	-	I	3.02	-	10.66	7.45 - 8.65	1.22 (bridging)	4.20 (bridging)	
{Ga ₂ (L ¹) ₃ }	-	-	3.04	-	10.70	6.82-8.56	-	-	
${AI(OPr^i) (L^2)}_2$	-	-	2.82	-	10.65	6.75 - 8.31	1.26(bridging)	4.12(bridging)	
${AI(L^2)_2}_2$	-	-	2.84	-	10.67	6.74 - 8.34	-	-	
{Ga(OPr ⁱ) (L ²)} ₂	-	-	2.88	-	10.72	6.92-8.42	1.20(bridging)	4.08 (bridging)	
$\{Ga_2(L^2)_3\}$	-	I	2.92	-	10.74	6.72-8.62	-	-	

Table 3

¹³C NMR spectral data (δ ppm) of the ligands and their aluminium (III) and gallium (III) complexes

Compound	Chemical Shift Values									
	>C=O / >C=N/		Aromatic carbons	Isopropox	(y group					
	>C=S	>C-N								
	180.03	167.15	160.72, 138.92, 130.15, 129.83 129.88 125.42,	-	-					
L^1H_2			122.57, 120.51, 118.77, 119.79							
L^2H_2	179.52	168.01	160.86, 138.99, 130.18, 129.79, 129.95, 125.58,	-	-					
			122.64, 120.68, 118.94, 119.99							
$\{AI(OPr^i) (L^1)\}_2$	181.92	168.05	160.54, 139.62, 131.15, 128.63 129.28 125.42,	73.16	23.81					
			122.57, 120.51, 118.77, 119.79	(bridging)	(bridging)					
$\{AI_2(L^1)_3\}$	182.12	169.02	160.72, 138.92, 130.15, 129.63 129.58 125.42,	-	-					
{AI2(L)3}			121.56, 121.51, 119.77, 118.79							
{Ga(OPr ⁱ) (L ¹)} ₂	182.54	169.07	160.48, 139.58, 130.24, 129.53 129.88 125.42,	74.86	24.92					
			120.57, 120.51, 118.76, 119.59	(bridging)	(bridging)					
$\{Ga_2(L^1)_3\}$	182.68	170.04	160.72, 138.92, 130.15, 129.83 129.88 124.42,	-	-					
{Ga2(L)3}			122.57, 121.51, 118.77, 119.79							
${AI(OPr^i) (L^2)}_2$	179.80	169.02	160.86, 138.99, 130.46, 129.79, 128.95, 123.58,	74.18	23.91					
			122.64, 120.68, 118.94, 118.98	(bridging)	(bridging)					
${AI(L^2)_2}_2$	180.04	169.81	160.72, 138.92, 130.15, 129.83 129.88 125.42,	-	-					
{ <i>/</i> ≺ı(∟ <i>)</i> 2}2			121.57, 120.51, 119.77, 119.79							

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	Ga(OPr ⁱ) (L ²)} ₂	180.12	169.53	160.54, 138.92, 130.15, 129.83 129.88	124.42,	73.19	23.24
ť	Ga(OPI)(L)			122.57, 120.51, 118.57, 119.79	(b	oridging)	(bridging)
	$\{Ga_2(L^2)_3\}$	180.60	170.03	160.62, 138.92, 130.15, 128.83 128.88	124.42,	-	-
	{Ga2(L)3}			122.57, 120.51, 118.77, 119.79			

Table 4

Fugicidal Screening Data for the Ligands and their Metal Complexes [Inhibition after 96 H (%) (Conc . In Ppm)]

Compound	Macrophomina phaseolina			Fusarium oxysporum			
	50	100	200	50	100	200	
L^1H_2	28±0.4	32±0.5	57±0.5	29±1.0	38 ±0.6	66 ±0.6	
L^2H_2	30 ±0.3	39 ±0.3	64±0.6	30 ±0.4	41±0.3	68 ±0.5	
{AI(OPr ⁱ) (L ¹)} ₂	29±0.08	41±0.5	62±0.4	29 ±0.8	42±0.5	67±0.4	
${AI(L^1)_2}_2$	30 ±0.6	44±0.2	65 ±0.5	31 ±0.4	47±0.8	68 ±0.5	
{Ga(OPr ⁱ)(L ¹)} ₂	30 ±0.3	42 ±0.4	66±0.5	32 ±0.5	43 ±0.6	68 ±0.3	
{Ga ₂ (L ¹) ₃ }	31±0.3	45±0.6	68±0.3	34±1.1	49 ±0.5	69 ±0.6	
{Al(OPri) (L2)}2	31±0.4	43±0.4	63±0.5	33±0.5	45±0.2	68±0.4	
${AI(L^2)_2}_2$	32±0.5	46±0.5	66±0.5	35 ±0.3	47±0.5	67 ±0.5	
{Ga(OPr ⁱ) (L ²)} ₂	33±0.7	43 ±0.4	68±0.3	36 ±0.4	48 ±0.3	70 ±0.6	
{Ga ₂ (L ²) ₃ }	34±1.1	46 ±0.4	70 ±0.7	30 ±0.4	49 ±0.6	74±0.5	
Flucanazole	82±0.8	100±0.4	100±1.2	86±0.5	100±0.4	100±0.6	



Antibacterial Screening Data of the Ligands and their Metal Complexes [Diameter of Inhibition Zone (Mm) (Concentration in Ppm)]

Compound	Staphyloc	occus aureus	Escher	richia col			
	500 ppm	1000 ppm	500 ppm	1000 ppm			
L^1H_2	5±0.02	7±0.08	4±0.05	6±0.08			
L^2H_2	6±0.04	8 ±0.07	6 ±0.02	8±0.02			
{AI(OPr ⁱ) (L ¹)} ₂	5 ±0.03	7 ±0.07	4±0.02	7±0.01			
${AI(L^1)_2}_2$	7±0.02	8 ±0.03	8±0.02	9 ±0.03			
${Ga(OPr^{i}) (L^{1})}_{2}$	6 ±0.05	8 ±0.05	6±0.02	9 ±0.02			
$\{Ga_2(L^1)_3\}$	8±0.1	9 ±0.05	9 ±0.09	10±0.02			
${AI(OPr^{I}) (L^{2})}_{2}$	8 ±0.05	9 ±0.05	7 ±0.03	9 ±0.01			
${AI(L^2)_2}_2$	9 ±0.06	10±0.05	9 ±0.03	10 ±0.01			
{Ga(OPr ⁱ) (L ²)} ₂	9 ±0.08	10 ±0.1	8±0.02	10 ±0.04			
$\{Ga_2(L^2)_3\}$	10±0.07	11 ±0.01	10±0.03	12±0.03			
Tetracycline	16±0.06	18 ±0.04	15±0.08	18±0.03			