

METAL COMPLEXES OF EGTA

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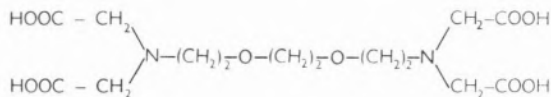
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Values valid for 20.0°C and $\mu = 0.1\text{ M}$ (KNO_3) are reported for the stability constants of 1:1 complexes, ML, and protonated complexes, MHL, formed by EGTA — ethyleneglycol (bis-aminoethylether) tetracetic acid — with a number of univalent and divalent ions (Ti^+ , Ag^+ , Mn^{2+} , Fe^{2+} , Co^{2+} , Ni^{2+} , Zn^{2+} and Pb^{2+}). Possible differences in the structure of these complexes are discussed on the basis of stability data.

1 — INTRODUCTION

SCHWARZENBACH has published data for a number of analogues of EDTA in which the chain separating the two chelating groups $-\text{N}(\text{CH}_2\text{COOH})_2$ is extended by additional methylene groups or additional methylene groups joined through symmetrically placed donor atoms (1).

One of the most interesting of these ligands is 1:2-bis-[2-di(carboxymethyl)aminoethoxy] ethane — as I — or ethyleneglycol(bis-aminoethylether)tetracetic acid, commonly abbreviated to EGTA, especially because the stability constants of its calcium and magnesium complexes differ so much that independent titration of the two metals is quite feasible (2).



I

EGTA became one of the complexones considered for analytical use and it is surprising that so few studies of its metal complexes have been carried up to the present; furthermore, most of the values reported in the literature were obtained using the mercury electrode (3) and protonated complexes were not taken into account.

In the present paper we extend the range of data for this complexone by reporting values for the stabilities

of its complexes with thallium (I), silver, manganese, iron, cobalt, nickel, zinc and lead.

2 — EXPERIMENTAL

2.1 — REAGENTS

1:2-bis- [2-di (carboxymethyl) aminoethoxy] ethane

The commercial G. Frederick Smith Chemical Co. product was dissolved in the equivalent amount of *NaOH* and precipitated with *AnalaR HCl*. The process was repeated and the purified acid was washed with water, ethanol and ether and dried at 60°C. Purity of the final product was over 99 % as determined by potentiometric titration.

Metal salts

All metal salts were of *AnalaR* grade; the concentration of the stock solutions was determined by complexometric titrations or other standard procedures.

Carbonate-free KOH

Carbonate-free *KOH*, concentration C_B , was prepared according to the instructions of SCHWARZENBACH and col. (4). Its ionic strength was made up to 0.100 M with potassium nitrate taking into account the neutralization of hydroxyl ions.

Water

All water used in this work was de-ionised, *e.g.*, distilled water which was passed through a mixed-bed ion-exchange resin.

2,2 — TECHNIQUE

Potentiometric titrations were carried out with a glass electrode (*Radiometer, type G 2025 B*) in a double-walled vessel maintained at 20.0°C with circulating water pumped from a thermostat bath.

The saturated calomel reference electrode was placed in a similar but smaller vessel connected to the first through a Laitinen bridge (5) of saturated KNO_3 .

The pH meter (*Radiometer pHM4*) was calibrated as a concentration probe by titrating perchloric, acetic and ethylenediaminetetracetic acids, and standardized each time with 0.05 M potassium hydrogen phthalate. The solutions to be titrated were made up by taking 100 ml of the solution of the ligand acid, concentration C_L , and adding 10 ml each of the solutions of the metal salts, concentration C_M , and of potassium nitrate, concentration such as to give a final ionic strength of 0.100 M. The initial total volume was then 120.0 ml.

A more detailed description is given elsewhere (6).

The results of the titrations are tabulated below. (Tables I, II, III).

3 — COMPUTATION OF STABILITY CONSTANTS

The method of computation was given before by IRVING and STACEY (7). From the equations for mass balance and electroneutrality, viz.,

$$C_L = [H_4L] + [H_3L^-] + [H_2L^{2-}] + [HL^{3-}] + [L^{4-}] + [ML^{2-}] + [MHL^-] \quad (1)$$

$$C_M = [M^{2+}] + [ML^{2-}] + [MHL^-] \quad (2)$$

$$[K^+] + [H^+] + 2[M^{2+}] = [H_3L^-] + 2[H_2L^{2-}] + 3[HL^{3-}] + 4[L^{4-}] + 2[ML^{2-}] + [MHL^-] + [X^-] + [OH^-] \quad (3)$$

where $[K^+] = a C_L$ is the concentration of potassium ions introduced during the titration and $[X^-] = 2 C_M$ is the concentration of the univalent anion arising from the salt of the bivalent metal M , we obtain

$$(4-a) C_L - [H^+] + [OH^-] = 4[H_4L] + 3[H_3L^-] + 2[H_2L^{2-}] + [HL^{3-}] + [MHL^-] \quad (4)$$

$$[M^{2+}] = C_M - C_L + \alpha_H [L^{4-}] \quad (5)$$

Table I

Reagent alone. $C_L = 9.56 \times 10^{-4} M$, $C_B = 9.88 \times 10^{-2} M$

KOH (v ml)	a	pH	KOH (v ml)	a	pH	KOH (v ml)	a	pH
0.00	0.000	2.884	1.30	1.344	3.400	2.60	2.687	8.807
0.10	0.103	2.911	1.40	1.447	3.473	2.70	2.791	8.902
0.20	0.207	2.937	1.50	1.550	3.568	2.80	2.894	8.990
0.30	0.310	2.966	1.60	1.654	3.692	2.90	2.997	9.069
0.40	0.414	3.001	1.70	1.757	3.852	3.00	3.101	9.144
0.50	0.517	3.029	1.80	1.861	4.166	3.10	3.204	9.200
0.60	0.620	3.070	1.90	1.964	4.900	3.20	3.308	9.285
0.70	0.724	3.102	2.00	2.067	7.226	3.30	3.411	9.344
0.80	0.827	3.142	2.10	2.170	7.978	3.40	3.514	9.422
0.90	0.931	3.184	2.20	2.274	8.278	3.50	3.618	9.496
1.00	1.034	3.222	2.30	2.377	8.451	3.60	3.721	9.590
1.10	1.137	3.277	2.40	2.481	8.600	3.70	3.824	9.677
1.20	1.240	3.331	2.50	2.584	8.712	3.80	3.928	9.768

a : number of equivalents of base added per mole of acid

and

$$[L^{4-}] = \frac{(4-a) C_L - [H^+] + [OH^-]}{\beta_H + [M^{2+}][H^+] K_{MHL}/k_4} \quad (6)$$

Where

$$\alpha_H = [H^+]^4/k_1 \cdot k_2 \cdot k_3 \cdot k_4 + [H^+]^3/k_2 \cdot k_3 \cdot k_4 + [H^+]^2/k_3 \cdot k_4 + [H^+]/k_4 + 1 \quad (7)$$

$$\beta_H = 4[H^+]^4/k_1 \cdot k_2 \cdot k_3 \cdot k_4 + 3[H^+]^3/k_2 \cdot k_3 \cdot k_4 + 2[H^+]^2/k_3 \cdot k_4 + [H^+]/k_4 \quad (8)$$

$$K_{ML} = [ML^{2-}]/[M^{2+}][L^{4-}] ;$$

$$K_{MHL} = [MHL^-]/[M^{2+}][HL^{3-}]$$

and

$$k_n = [H^+][H_{4-n}L^{n-}]/[H_{5-n}L^{1-n}] .$$

K_{ML} and K_{MHL} can be evaluated by successive approximations starting with $K_{MHL} = 0$; this enables $[L^{4-}]$ to be calculated and from this value a series of simultaneous equations of the type

$$\bar{n} + (\bar{n}-1)[H^+][L^{4-}] K_{MHL}/k_4 + (\bar{n}-1)[L^{4-}] K_{ML} = 0 \quad (9)$$

Table II

REAGENT IN THE PRESENCE OF VARIOUS METALS

$C_L = 9.56 \times 10^{-4} M$; $C_B = 9.88 \times 10^{-2} M$;
 C_M as indicated below

KOH (v ml)	<i>a</i>	pH			
		Tl^+	Ag^+	Mn^{2+}	Co^{2+}
0.00	0.000	2.887	2.881	2.874	2.861
0.20	0.207	2.943	2.926	2.920	2.903
0.40	0.414	2.997	2.981	2.974	2.953
0.60	0.620	3.059	3.042	3.037	3.007
0.80	0.827	3.130	3.111	—	3.066
1.00	1.034	3.218	3.194	3.200	3.137
1.20	1.240	3.321	3.292	3.296	3.218
1.40	1.447	3.462	3.420	3.429	3.312
1.60	1.654	3.664	3.607	3.613	3.422
1.80	1.861	4.067	3.915	3.846	3.553
1.90	1.964	4.538	4.257	4.009	3.627
2.00	2.067	6.364	5.030	4.128	3.711
2.10	2.170	7.190	6.181	4.216	3.811
2.20	2.274	7.531	6.550	4.305	3.923
2.30	2.377	7.755	6.745	4.385	4.039
2.40	2.481	7.941	6.907	4.462	4.159
2.50	2.584	8.096	7.035	4.533	4.272
2.60	2.687	8.242	7.143	4.595	4.391
2.70	2.791	8.369	7.261	4.666	4.512
2.80	2.894	8.495	7.367	4.738	4.628
2.90	2.997	8.616	7.468	4.812	4.741
3.00	3.101	8.730	7.576	4.891	4.860
3.10	3.204	8.850	7.686	4.979	4.974
3.20	3.308	8.953	7.818	5.091	5.090
3.30	3.411	—	7.969	5.228	5.220
3.40	3.514	9.177	8.158	5.381	5.353
3.50	3.618	9.289	8.392	5.560	5.497
3.60	3.721	9.403	8.596	5.771	5.660
3.70	3.824	9.525	8.791	6.018	5.847
3.80	3.928	9.650	8.984	6.370	6.110
3.90	4.031	9.785	9.151	7.088	6.504
C_M		0.0105	0.0100	0.0097	0.0097

where

$$\bar{n} = (C_L - \alpha_H [L^{4-}]) / C_M \quad (10)$$

can be solved, giving a new value for K_{MHL} and a first approximation for K_{ML} .

The method is repeated until satisfactory convergency of values is obtained.

Table III

REAGENT IN THE PRESENCE OF VARIOUS METALS

$C_L = 9.83 \times 10^{-4} M$; $C_B = 9.88 \times 10^{-2} M$;
 C_M as indicated below

KOH (v ml)	<i>a</i>	pH			
		Fe^{2+}	Ni^{2+}	Zn^{2+}	Pb^{2+}
0.00	0.000	2.882	2.799	2.848	2.696
0.20	0.201	2.934	2.825	2.901	2.724
0.40	0.402	2.990	2.863	2.955	2.758
0.60	0.603	3.048	2.904	3.020	2.793
0.80	0.804	3.112	2.951	3.084	2.833
1.00	1.005	3.192	3.001	3.147	2.877
1.20	1.206	3.285	3.053	3.230	2.925
1.40	1.407	3.404	3.113	3.314	2.980
1.60	1.608	3.560	3.188	3.415	3.040
1.80	1.809	3.760	3.271	3.558	3.110
1.90	1.910	3.887	3.319	3.631	3.151
2.00	2.010	4.037	3.360	3.712	3.193
2.10	2.111	4.158	3.417	3.798	3.242
2.20	2.211	4.269	3.485	3.885	3.271
2.30	2.312	4.370	3.558	3.982	3.330
2.40	2.412	4.459	3.650	4.090	3.400
2.50	2.513	4.542	3.753	4.203	3.487
2.60	2.613	4.622	3.886	4.320	—
2.70	2.714	4.700	4.055	4.445	3.718
2.80	2.814	4.774	4.280	4.562	3.871
2.90	2.915	4.850	4.577	4.679	4.075
3.00	3.015	4.929	4.897	4.801	4.320
3.10	3.116	5.004	5.191	4.930	4.552
3.20	3.216	5.092	5.431	5.061	4.757
3.30	3.317	5.192	5.661	5.201	4.939
3.40	3.417	5.300	5.872	5.360	5.111
3.50	3.518	5.421	6.101	5.530	5.300
3.60	3.618	5.570	6.329	5.742	5.510
3.70	3.719	5.761	6.568	6.052	5.746
3.80	3.819	6.016	6.848	6.660	6.020
3.90	3.920	6.544	7.308	9.007	6.637
4.00	4.020	7.212	8.100	9.710	9.151
C_M		0.0096	0.0100	0.0099	0.0100

An electrical desk calculating machine was used for the calculations and convergency was considered satisfactory when successive approximations gave values not differing more than ± 0.01 log units.

Six to eight experimental points were used in the computations and the best fitting lines were determined by a least squares method.

4 — RESULTS AND DISCUSSION

The titration curves (fig. 1) show that protonated complexes must be taken into account and that their stability is appreciable compared to that of the normal complex because pK_3 for the ligand acid is of the order of pK_4 .

Higher complexes and polynuclear complexes should not occur under the experimental conditions used; however, the titration data for copper did not give consistent results, certainly because M_2L complexes of high stability are formed.

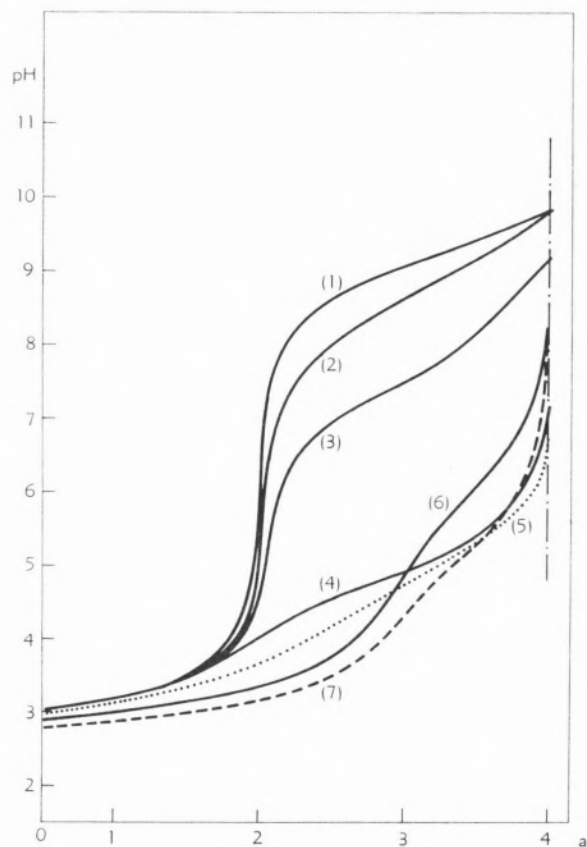


Fig. 1 — Titration curves for EGTA alone and in the presence of various univalent and divalent ions

$T = 20^\circ\text{C}$, $\mu = 0.1 \text{ M (KNO}_3\text{)}$

1 — Free ligand ; 2- Tl^+ ; 3- Ag^+ ; 4- Fe^{2+} ;
5- Co^{2+} ; 6- Ni^{2+} ; 7- Pb^{2+} .

Although this fact was recently contradicted by polarographic studies (8), we have confirmed it beyond any doubt with spectrophotometric evidence (fig. 2).

The Job curves in fig. 2 clearly show that 1:1 and 2:1 complexes coexist at pH 4.0. The 2:1 complex

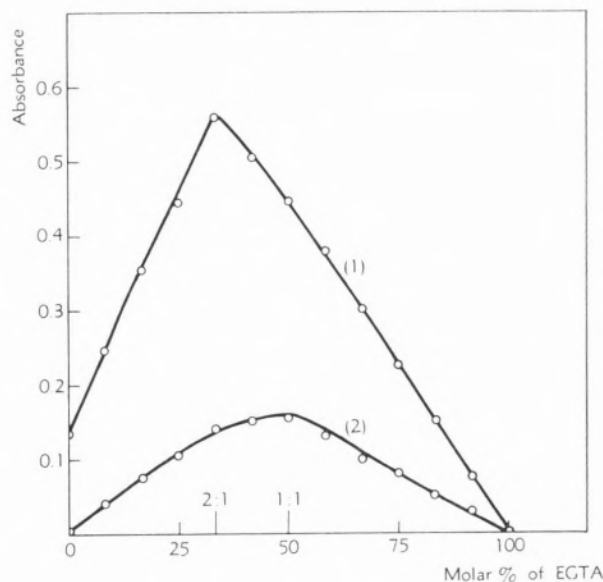


Fig. 2 — Method of the continuous variations for copper-EGTA complexes

Total concentration $C_M + C_L = 1.44 \times 10^{-2} \text{ M}$; $\text{pH} = 4.0 - 4.5$

(1) $\lambda = 750 \text{ m}\mu$; (2) $\lambda = 580 \text{ m}\mu$.

absorbs at $\lambda_{\text{max}} = 735 \text{ m}\mu$ and the normal 1:1 complex at about $570 \text{ m}\mu$. For 1:1 mixtures of Cu^{2+} and EGTA the absorption curve shows two shoulders corresponding to both complexes, thus supporting our view on the reason for the lack of convergency in the calculations. Tables IV and V summarise the values obtained for the dissociation constants of EGTA and for the stability constants of its metal complexes, together with other values obtained by previous workers.

Table IV

DISSOCIATION CONSTANTS OF EGTA

	(a)	(b)
pK_1	< 2	~ 2
pK_2	2.65	2.68
pK_3	8.78	8.85
pK_4	9.46	9.46

(a) Present work. $T = 20.0^\circ\text{C}$; $\mu = 0.1 \text{ M (KNO}_3\text{)}$

(b) Ref.⁸ 1 : $T = 20.0^\circ\text{C}$; $\mu = 0.1 \text{ M (KCl)}$

Agreement with the values obtained by HOLLOWAY and REILLEY (3) is quite satisfactory, minor deviations being due to the consideration of protonated complexes in this work. Values recently presented by RINGBOM (9) differ considerably from ours and from those of the previous workers; the reason for the discrepancies cannot be ascertained.

complexes, contrarily to what happens in the protonated complexes. The reason for this inversion cannot be unambiguously explained but it is likely that it results from different structures for the normal complexes as suggested below.

Comparison of the values of stability constants of the complexes of EGTA with the correspondent values for

Table V

STABILITY CONSTANTS OF METAL COMPLEXES OF EGTA

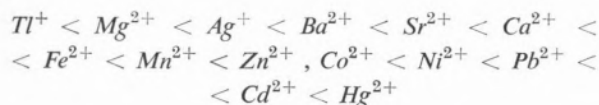
 $T = 20.0^\circ\text{C}$

Ion	$\log K_{MHL}^M$		$\log K_{ML}$				$\text{p}K_{MHL}^H$		
	(a)	(b)	(a)	(b)	(c)	(d)	(a)	(b)	(c)
Ag^+	4.93		6.88				7.51		
Ba^{2+}		4.26		8.41				5.31	
Ca^{2+}		5.33		11.00	10.9			3.8	
Cd^{2+}		10.72		16.73		15.6			3.5
Co^{2+}	7.99		12.50		12.30		4.95		
Cu^{2+}					17.8	17			4.4
Fe^{2+}	6.93		11.92				4.47		
Hg^{2+}		16.76		23.20	23.8			3.0	
Mg^{2+}		3.37		5.21				7.6	
Mn^{2+}	6.59		12.11		12.3	11.5	3.94		5.0
Ni^{2+}	9.19		13.55		13.6	12.0	$6.13 \pm 0.03^*$		6.0
Pb^{2+}	10.28		14.71		14.6	13.0	$4.96 \pm 0.02^*$		5.3
Sr^{2+}		4.37		8.50				5.3	
Tl^+	3.85		4.38				8.93		
Zn^{2+}	7.97		12.49			12.8	4.94		5.2

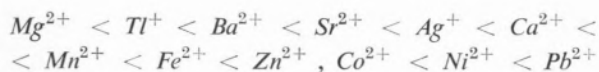
(a) Present work : $\mu = 0.1 \text{ M (KNO}_3\text{)}$ (b) Ref.⁶ 1 : $\mu = 0.1 \text{ M (KCl)}$ (c) Ref.⁶ 3(d) Ref.⁶ 9

* Direct potentiometric determination.

The order of stabilities for the normal ML complexes of EGTA is



And for the protonated complexes,



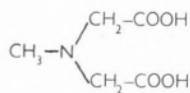
The IRVING-WILLIAMS order of stabilities (10) is not followed by the pair Mn^{2+} , Fe^{2+} in their normal

EDTA and EEDTA (ethyletherdiaminetetracetic acid) is presented in graphical form (fig. 3).

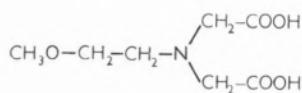
The behaviour of EEDTA is close to that of EDTA, as commented before (1); EGTA, however, behaves differently: the stability of the complexes of calcium and barium is higher than that of the corresponding complexes of the other ligands, whereas for magnesium the well-known lowering in stability is verified. What is not so frequently realized is that for the transition metals identical lowering of stability occurs, and this cannot be just the result of differences in ionic radii; different structures for these complexes must then be postulated.

To help the discussion of this problem we have sum-

marized in Table VI the data for the complexes of methyliminodiacetic acid (II) and methoxyethyliminodiacetic acid (III).



II



III

The values of $\log K_{MHL}$ for the complexes of *Fe*, *Co*, *Ni* and *Zn* with EGTA are all of the same order of magnitude of the correspondent values for the normal com-

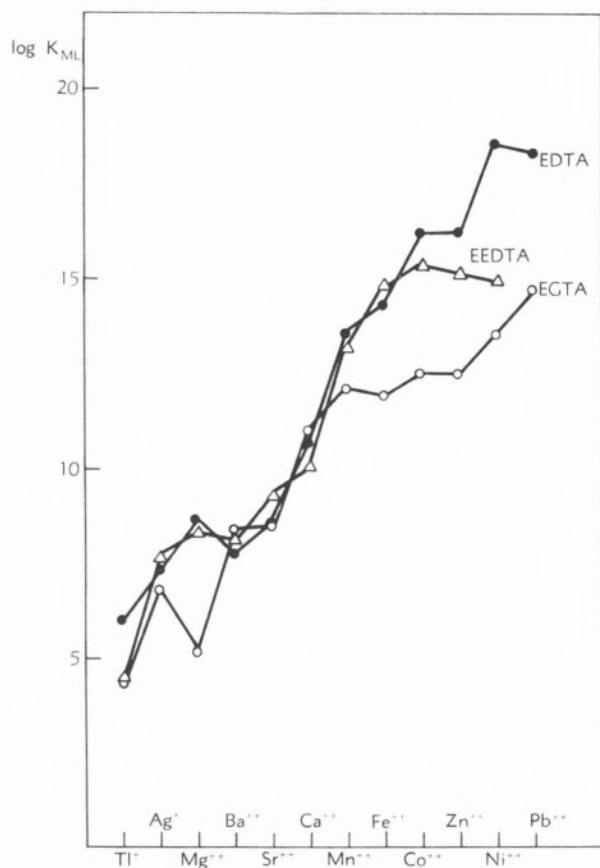
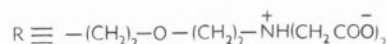
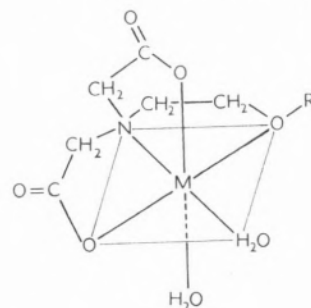


Fig. 3 — Comparison of the stabilities of metal complexes of EGTA (open circles), EDTA (filled circles) and EEDTA (triangles). Data valid for $T = 20^\circ\text{C}$ and $\mu = 0.1\text{M}$ from the present work and references 11 and 12 (values for *Co*, *Zn* and *Ni* complexes with EEDTA)

plexes of methoxyethyliminodiacetic acid (III), and it is likely that both types of complexes have analogous structures, possibly as in IV.

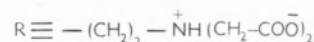
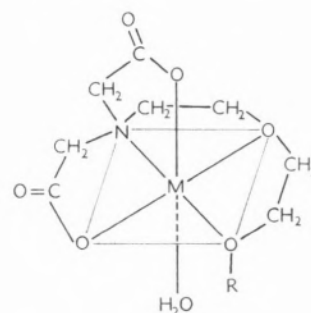
The fact that copper and possibly the other transition metals form 2:1 complexes supports this formulation. In the case of *Mg* the values of stability constants for



IV

the complexes of the three ligands suggest that in the protonated complex of EGTA only one iminodiacetate group is coordinated.

For the other alkaline earth metals and also for *Cd*, *Hg*, *Mn* and *Pb* the values of $\log K_{MHL}$ of EGTA complexes are considerably higher than the values of $\log K_{ML}$ for the complexes of methoxyethyliminodiacetic acid. This cannot be due to the tendency of all these metals for tetrahedral arrangements and must imply coordination by the second ether oxygen atom in the molecule of EGTA as in V.



V

If the stability constants of the normal *ML* complexes of EGTA are compared with the overall stability constants for the complexes of the other ligands in Table VI, the same results are obtained, e.g., the complexes formed by *Fe*, *Co*, *Ni*, *Zn* and possibly *Cu* are all less stable

than the ML_2 complexes formed by methyliminodiacetic acid and methoxyethyliminodiacetic acid.

The opposite is verified for the complexes of all the other metals which were considered.

maining metals, which prefer coordination to oxygen atoms, structure VII, deriving from V, is suggested.

Structure VI had already been proposed for the complex formed by the Fe^{3+} ion¹³. This agrees with our hypo-

Table VI

	EGTA		Methyliminodiacetic ^d acid		Methoxyethyliminodiacetic ^d acid	
	I		II		III	
	$\log K_{MHL}$	$\log K_{ML}$	$\log K_{ML}$	$\log \beta_{ML_2}$	$\log K_{ML}$	$\log \beta_{ML}$
H^+	8.78 (a)	9.46 (a)	9.65		8.96	
Ba^{2+}	4.26 (b)	8.41 (b)	2.59		3.56	
Ca^{2+}	5.33 (b)	10.9 (c)	3.75		4.53	
Cd^{2+}	10.72 (b)	16.73 (b)	6.77	12.52	7.53	13.18
Co^{2+}	7.99 (a)	12.50 (a)	7.62	13.91	7.96	12.90
Cu^{2+}		17.8 (c)	11.09	17.92	12.34	16.59
Fe^{2+}	6.93 (a)	11.92 (a)	6.65	12.02	6.81	10.73
Hg^{2+}	16.76 (b)	23.20 (b)	5.47	9.15	5.94	10.03
Mg^{2+}	3.37 (b)	5.21 (b)	3.44		3.31	
Mn^{2+}	6.59 (a)	12.11 (a)	5.40	9.56	5.53	9.62
Ni^{2+}	9.19 (a)	13.55 (a)	8.73	15.95	9.39	14.85
Pb^{2+}	10.28 (a)	14.71 (a)	8.02	12.12	9.49	13.24
Sr^{2+}	4.37 (b)	8.50 (b)	2.85		3.84	
Zn^{2+}	7.97 (a)	12.49 (a)	7.66	14.09	8.43	12.85

(a) Present work.

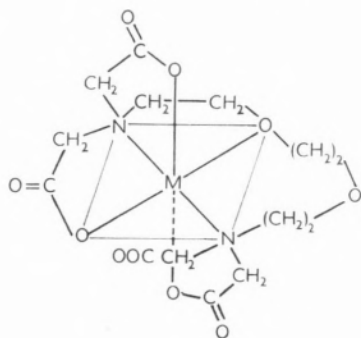
(b) Ref.^o 1.

(c) Ref.^o 3.

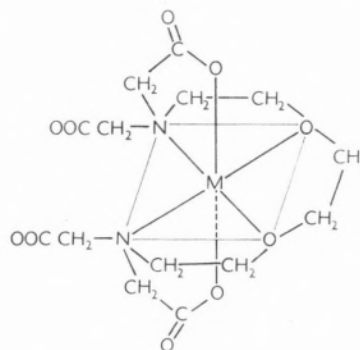
(d) Ref.^o 11.

Since the transition metals have considerable tendency to coordinate with a nitrogen atom rather than with

thesis, because the ferric ion also prefers to coordinate with oxygen atoms.



VI

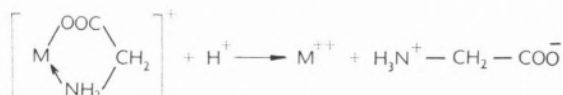


VII

an oxygen atom, these observations are compatible with structure VI, deriving from IV, for the complexes of Fe , Co , Ni , Cu and Zn . For the complexes of the re-

Studies with molecular models show that both structures VI and VII are possible, thus supporting our suggestion.

Finally, if structure VI is correct, one would expect that the reaction of protonation of this species is somewhat analogous to the reaction.



e.g. to the protonation of glycine complexes (7).

The change in GIBBS free energy in the latter reaction is measured by $\text{pk}_{\text{HX}} - \log K_{\text{MX}}$ ($X \equiv$ glycine), and in the first reaction (VI \rightarrow IV) is measured by pk_{MHL} . If the reactions are analogous $\text{pk}_{\text{HX}} - \log K_{\text{MX}} \simeq \text{pk}_{\text{MHL}}$. For Co and Zn the calculated values, *e.g.*, the values $\text{pk}_{\text{HX}} - \log K_{\text{MX}}$, are 4,96 and 4,66, and the experimental values are, respectively, 4,96 and 4,95, in excellent agreement.

For Fe and Ni, however, the differences between calculated and experimental values are greater, showing that this comparison is probably too simple or that other types of structure must be considered.

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RESUMO

Apresentam-se valores de constantes de estabilidade dos complexos ácidos e normais do EGTA — ácido etilenoglicol (bis-aminoetiléter) tetracético — formados com uma série de iões mono e divalentes (Ti^+ , Ag^+ , Mn^{2+} , Fe^{2+} , Co^{2+} , Ni^{2+} , Zn^{2+} e Pb^{2+}). Com base nestes valores discutem-se possíveis diferenças na estrutura dos respectivos complexos.