blue-green solution. The mixture was stirred magnetically for 1 hr. at 0.0° without any noticeable change. The trimethylamine was removed by distillation to another trap on the vacuum line, where it was kept at a temperature slightly below 0° so that its vapor, at an equilibrium pressure of 635 mm., was in contact with the copper(II) chloride in the reaction tube. The reaction tube was then heated in a water bath to 85°. Very suddenly the brown solid copper(II) chloride absorbed trimethylamine, causing the pressure in the system to drop and amine to distil back into the reaction tube. The product was a clear amber oil. Any excess of trimethylamine swept into the reaction tube was removed by pumping at room temperature; the gain in weight of the tube corresponded to the retention of 11.0 mmoles of trimethylamine in the reaction product; mole ratio 1.14.

When 1 ml. of tri-*n*-butylamine was added to a mixture of 1 g. of copper(II) chloride and 10 ml. of dimethyl sulfoxide, a deepening of the green color of the system was observed, and on warming a dark red oil slowly separated. On addition of water to the oil, a precipitate of copper(I) chloride was formed. Tri-*n*-butylamine caused precipitation of hydrous oxide when added to a solution of copper(II) chloride in 95% alcohol, and no reaction was observed on boiling this mixture for several minutes. When absolute alcohol was used, the solution darkened on addition of the amine, and on warming slowly took on a dark reddish brown opaque appearance.

Addition of N-methylaniline or N,N-dimethylaniline to a solution of copper(II) chloride in absolute alcohol led to immediate formation of violet to jet black solid products.

Reaction of Oxygen with the Copper(II) Chloride–Triethylamine Reaction Product.—When oxygen gas was bubbled through the red solution obtained by the reaction of triethylamine with copper(II) chloride in mole ratio 2:1 in absolute alcohol, there was little change in the color of the solution but a precipitate was slowly formed. The dull green precipitate was removed by filtration and found, in the manner described above, to be mostly copper(I) chloride. Some oxidation to copper(II) had apparently occurred, but the major oxidative action was on the organic substance holding the copper(I) in solution. Evaporation of the filtrate gave a reddish product from which triethylammonium chloride was leached and identified as described previously.

Triethylammonium Dichlorocuprate(I)–Triethylamine System.—The liquid chlorocuprate(I) complex obtained from 5.03 mmoles of copper(I) chloride and 5.09 mmoles of triethylammonium chloride in a tube on the vacuum system was treated with triethylamine, and the excess of triethylamine was removed by distillation at 25° in the vacuum system. A sharp drop in pressure occurred at the point where 5.02 mmoles of triethylamine was retained in the reaction tube. The product was a white crystalline solid with a dissociation pressure of 6 mm. Triethylamine was then removed at this equilibrium pressure, this being accompanied by conversion of the solid back to an oil.

Triethylammonium Dichlorocuprate(I)–Tri-*n*-butylamine System.—Similarly, an excess of tri-*n*-butylamine was distilled in the vacuum system (vapor pressure < 1 mm. at 25°) onto the oil formed from triethylammonium chloride and copper(I) chloride. The system was not completely homogeneous. Volatile material was removed at an equilibrium pressure of 8 mm. at 25° and identified as triethylamine by conversion to the hydrochloride; m.p. and mixture m.p. (sealed tube) 254°.

Contribution From The Kedzie Chemical Laboratory,
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Complexes of Some 1-Substituted Tetrazoles and Cobalt(II), Nickel(II), Platinum(II), and Zinc(II) Chlorides

By G. L. GILBERT and C. H. BRUBAKER, Jr.

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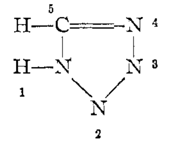
The compounds formed when zinc chloride, nickel chloride, and platinum(II) chloride react with 1-methyltetrazole, 1-cyclohexyltetrazole, and 1-phenyltetrazole in ethanol or tetrahydrofuran have the general formula MT_2Cl_2 (where T = 1-substituted tetrazole). The solids, with the exception of the zinc compounds, are insoluble in common solvents and decompose without melting upon heating. The complexes formed with zinc chloride are moderately soluble in tetrahydrofuran and ethanol and crystallize in clear, well-formed crystals from ethanol. Stability constant measurements with nickel in ethanol and cobalt in tetrahydrofuran yield similar values for the formation constants with 1-methyl- and 1-cyclohexyltetrazole.

Solid compounds resulting from the interaction of 5-substituted tetrazoles with silver, copper, iron(II), and cobalt have been reported[1-4] and the evidence indicated high stability due to anion formation by the tetrazole ring. Very little interaction occurs when both the 1 and 5 positions on the tetrazole are blocked. Examples of the formation of solid materials with 1-substituted tetrazoles are limited to the compound between platinum(IV) chloride and 1-ethyltetrazole.[5] A formal similarity of the tetrazole ring to cyclopentadiene has prompted the further investigation of these ring systems. The present work concerns the preparation of solid complexes between zinc chloride, nickel chloride, and platinum(II) chloride and certain 1-substituted tetrazoles and studies of cobalt(II) and nickel(II) complexes in solution.



Experimental

Materials.—Reagent grade chemicals were used throughout this investigation. The tetrazoles used were prepared by the method of Herbst and Fallon,[6] in the case of 1 phenyltetrazole, or

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(1) N. A. Daugherty, Ph.D. Thesis, Michigan State University, 1961.

(2) N. A. Daugherty and C. H. Brubaker, *J. Am. Chem. Soc.*, **83**, 3779 (1961).

(3) C. H. Brubaker, Jr., *ibid.*, **82**, 82 (1960).

(4) H. Jonassen, J. Terry, and A. Harris, private communication.

(5) E. Oliveri-Mandala and B. Alagna, *Gazz. chim. ital.*, **40**, II, 441 (1910).

(6) F. Fallon and R. M. Herbst, *J. Org. Chem.*, **22**, 933 (1957).

TABLE I

ANALYSES OF THE MT_2Cl_2 COMPOUNDS OF ZINC, PLATINUM, AND NICKEL

Compound	Found					Calculated				
	M	Cl	C	N	H	M	Cl	C	N	H
1-Methyltetrazoles										
$ZnCl_2C_4N_8H_8$	21.6	23.4	15.8	36.7	2.6	21.5	23.3	15.8	36.8	2.6
$PtCl_2C_4N_8H_8$	45.0	16.4	15.5	26.0	1.6	44.9	16.4	16.3	25.8	1.9
$NiCl_2C_4N_8H_8$	19.7	23.6	16.1	37.6	2.6	19.7	23.8	16.1	37.6	2.7
1-Cyclohexyltetrazoles										
$ZnCl_2C_{14}H_8H_{24}$	14.8	16.1	38.2	25.4	5.5	14.9	16.1	38.1	25.4	5.4
$PtCl_2C_{14}N_8H_{24}$	34.2	12.3	29.4	19.7	4.2	34.9	12.3	29.5	19.6	4.2
$NiCl_2C_{14}N_8H_{24}$	13.5	15.8	38.3	24.7	6.1	13.5	16.3	38.7	25.8	5.5
1-Phenyltetrazoles										
$ZnCl_2C_{14}N_8H_{12}$	15.1	16.5	39.3	26.2	2.8	15.2	16.7	39.2	26.1	2.8

by reaction of an 8% solution of hydrazoic acid in toluene with the isocyanide for 1-methyl- and 1-cyclohexyltetrazole. The isocyanides were produced from their formamides by the method of Ugi and Meyr for methylisocyanide[7] and the procedure of Ugi, *et al.*, for cyclohexylisocyanide.[8]

The tetrazoles were purified by sublimation under reduced pressure. Purity was confirmed by comparing melting points and infrared spectra with literature values.

Platinum(II) chloride was prepared by reduction of H_2PtCl_6 with hydrazine hydrochloride.[9] Nickel chloride was prepared by dehydration of the hexahydrate by reaction with thionyl chloride and subsequent removal of the remaining water under vacuum. *Anal.* Calcd. for $NiCl_2$: Cl, 54.7. Found: Cl, 54.7. Reagent grade $ZnCl_2$ was used without further treatment.

Analytical Methods.—Zinc was determined potentiometrically in an aqueous medium containing $Fe(CN)_6^{-3}$ with 0.1 N hexacyanoferrate(II) using a calomel and platinum electrode system.[10] The nickel content was determined by the procedure of Daugherty.[1] Platinum was determined by ignition at 600°. Chloride was determined potentiometrically in 0.1 N nitric acid with 0.1 N $AgNO_3$ using a calomel and silver–silver chloride electrode system. Analyses for carbon, hydrogen, and nitrogen were carried out by Spang Microanalytical Laboratory.

Spectrophotometric measurements were made using a Beckman Model DU spectrophotometer.

Preparation of the Metal Chloride–Tetrazole Complexes.—The solid complexes were prepared by adding the tetrazole (0.02 mole) to 75 ml. of tetrahydrofuran, freshly distilled from calcium hydride, in a 100-ml. round-bottomed flask. The metal chloride (0.01 mole) was added to a Soxhlet cup and the tetrazole solution was refluxed overnight to extract the metal as the tetrahydrofuran complex[11]; the tetrazole complexes formed in the bottom flask as the metal halide was extracted. Due to the low solubility of nickel chloride in tetrahydrofuran, 95% ethanol was used as the extraction solvent in order to obtain larger yields of the nickel complex. Infrared spectra of the nickel complexes, made in either ethanol or tetrahydrofuran, were identical.

After the solutions were cooled and filtered, the solvent was removed by pumping with a water aspirator. The solid was dried at 60° under vacuum and analyzed. Analytical data for the complexes are given in Table I.

Attempts to prepare cobalt(II) compounds resulted in solids of indefinite composition.

The analysis for chloride and hydrogen in the nickel 1-phenyl compound and for carbon in the platinum 1-methyl compound are farther from the expected values than one might hope. Because of thermal instability, carbon and hydrogen analyses of the compounds were difficult and frequently had to be repeated several times until suitable heating procedures were developed.

Infrared Spectra of the Complexes.—The infrared spectra of the free tetrazoles, the tetrazole hydrochlorides, and the metal chloride–tetrazole complexes prepared were obtained with KBr disks. Minor shifts in certain absorption maxima are observed, but no definite pattern to the shifts was determined. The absorbance at 1470 cm.$^{-1}$, attributed to ring C–H by Fallon,[12] is not shifted and indicates no great change in the ring due to coordination. The data are given in Table II.

Results and Discussion

The crystalline complexes $Zn(C_2N_4H_4)_2Cl_2$, $Zn(C_7N_4H_{12})_2Cl_2$, $Zn(C_7N_4H_6)_6Cl_2$, $Ni(C_2N_4H_4)_2Cl_2$, $Ni(C_7N_4H_{12})_2Cl_2$, $Pt(C_2N_4H_4)_2Cl_2$, and $Pt(C_7N_4H_{12})_2Cl_2$ have been prepared and characterized. The nickel and platinum compounds were found to be insoluble in common solvents and all decompose without melting upon heating. The zinc complexes are moderately soluble in ethanol and tetrahydrofuran.

The formation constants for nickel with 1-methyltetrazole and 1-cyclohexyltetrazole in absolute ethanol and for cobalt with 1-methyltetrazole and 1-cyclohexyltetrazole in tetrahydrofuran have been determined spectrophotometrically. The absorbancies, $\bar{n}$, and concentrations are listed in Tables III and IV.

The wave lengths selected for the study correspond to maxima in the spectra of either the complex or the uncomplexed metal. The nickel salt has maxima at 370 and 580 mμ, the complex at 420 mμ and a weaker broad band between 650 and 800 mμ. The cobalt chloride absorbs at 605 mμ and intensity falls and the band broadens as complexing occurs.

Limiting absorbancies were obtained by adding a large excess of tetrazole to the metal chloride solution. The degree of formation, α, may be calculated from the expression $\alpha = (A - A_0)/(A_\infty - A_0)$ or $\alpha = (A_0 - A)/(A_0 - A_\infty)$. The latter expression is used when the absorbancy of the complex is lower than that of the solvated metal salt. A is the absorbancy of any solution containing metal chloride and tetrazole, A_0 is the absorbancy of uncomplexed 0.001 M Co^{+2} or 0.01 M Ni^{+2}, and A_∞ is the limiting absorbancy.

Thus Bjerrum's[13] $\bar{n}$ is given by

$$\bar{n} = 2\alpha = 2(A - A_0)/(A_\infty - A_0)$$

or

$$\bar{n} = 2\alpha = 2(A_0 - A)/(A_0 - A_\infty)$$

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[7] I. Ugi and R. Meyr, *Ber.*, **93**, 239 (1960).

[8] I. Ugi, R. Meyr, M. Lipenshi, F. Bodesheim, and F. Rosendahl, *Org. Syn.*, **41**, 31 (1961).

[9] W. Cooley and D. Busch, *Inorg. Syn.*, **5**, 208 (1957).

[10] H. Willard, L. Merrit, and J. Dean, "Instrumental Methods of Analysis," D. Van Nostrand, Inc., Princeton, N. J., 1958.

[11] W. Herwig and H. H. Zeiss, *J. Org. Chem.*, **23**, 1404 (1958).

[12] F. Fallon, Ph.D. Thesis, Michigan State University, 1956.

[13] J. Bjerrum, "Metal Ammine Formation in Aqueous Solution," P. Haase and Son, Copenhagen, 1957.

TABLE II

INFRARED ABSORPTION BANDS (IN CM.$^{-1}$) OF 1-SUBSTITUTED TETRAZOLE, THE HYDROCHLORIDE, DICHLOROBIS-(TETRAZOLE)-ZINC(II), DICHLOROBIS-(TETRAZOLE)-NICKEL(II), AND DICHLOROBIS-(TETRAZOLE)-PLATINUM(II) COMPOUNDS; POTASSIUM BROMIDE DISKS

1-Methyltetrazole

$C_2N_4H_4$	$C_2N_4H_4 \cdot$ HCl	$Zn(C_2N_4H_4)_2Cl_2$	$Ni(C_2N_4H_4)_2Cl_2$	$Pt(C_2N_4H_4)_2Cl_2$
3401 vs		3509–3390 ms		3401 m
3100 vs		3125 s	3096 s	3077 s
2900 m				
1704 vw		1770–1751 vw	1754 w	1730 vw
1637 m		1610 vw	1621 m	1626–1613 w
1497 s		1524 s	1506 s	1517 s
1471 ms	1471 s	1471 m	1460 ms	1460 m
1418		1441 m	1412 m	
1277 m	1266 w	1311 m	1290 ms	1299 ms
1227 w		1236 w	1235 w	1247 w
1170 s	1167 s	1183 s	1171 s	1183 s
			1126 ms	
1109 s		1104 s	1105 s	1099 s
1083 w	1078 vw	1074 w	1074 w	1070 m
1053 vw		1027 m	1022 m	1020 m
1018 vw		1012 ms		
		996 s	993 s	
966 ms	959 s	890 s	880 s	889 w
877 w	869 s	882 s		862 s
			755–749 w	
		717 m	718 m	709 m
720 ms	719 m			

1-Phenyltetrazole

$C_7H_4N_6$	$C_7N_4H_6 \cdot HCl$	$Zn(C_{14}N_8H_6)$
3436 m	3367 m	3497 w
3025 ms	3077 ms	3077 s
1605 m	1592 ms	1600 ms
1504 s	1490 s	1511 s
1471 m	1460 ms	1473 m
1397 mw	1389 m	1416 ms
1337 vw	1330 vw	
		1311 w
		1292 w
1280 vw	1272 w	
1211 s		1221 s
1196 m	1198 s	
1181 s	1186 m	1185 ms
	1172 m	1176 ms
1096 s		
1083 mw	1086 s	1088 s
	1075 w	
1053 m		1053 m
		1008 s
1000 ms	993 ms	1000 m
966 m	962 m	985 w
919 ms	913 m	917 w
886 m	881 mw	896 mw
		832 w
762 s	756 s	763–760 s
717 w	713 w	712 mw

1-Cyclohexyltetrazole

$C_7N_4H_{12}$	$C_7N_4H_{12} \cdot$ HCl	$Zn(C_7N_4H_{12})_2$-Cl_2	$Ni(C_7N_4H_{12})_2$-Cl_2	$Pt(C_7N_4H_{12})_2$-Cl_2
			3322 s	
3100 m	3096 m	3058 m	3067 m	3077 vs
2899 s	2890 s	2915 s	2899 s	2882 vs
		2849 m	2833 m	2833 s
1672 m		1639 w	1634 m	
		1493 s	1488 m	1490 ms
1470 s	1464 m	1464 w		
	1441 s	1449 s	1449 s	1439 s
1366 w	1346 w	1368 s	1366 w	1366 w
		1346 w		
		1298 w		1290 m
1266 w	1264 w	1266 w		
1167 s	1167 s	1168 s	1174 s	1167 s
1139 m	1134 ms	1135 s	1136 m	1133 m
1101 s	1096 s	1089 s	1093 s	1081 s
			1079 w	
1054 w		1052 w		
1030 w	1027 vw	1014 s	1015 s	1024 ms
	999 ms			989 w
970 m	969 w	966 vvw		
897 w	895 w	893 ms	893 ms	892 m
881 w	879 s	876 m		868 m
818 w	816 w	816 m	814 mw	810 w
749 m	746 ms	743 ms	754 s	743 ms
		715 w		

TABLE III

ABSORBANCIES AND $\bar{n}$ VALUES FOR 1-SUBSTITUTED TETRAZOLES AND 0.01 *M* NICKEL CHLORIDE HEXAHYDRATE IN ABSOLUTE ETHANOL

[L], *M*	− log [L]	*A*420 mμ	$\bar{n}$
1-Methyltetrazole			
0.00	...	0.107	0.000
.03	1.52	.094	.313
.04	1.40	.090	.410
.05	1.30	.085	.530
.06	1.22	.079	.675
.07	1.15	.074	.795
.08	1.10	.070	.892
.09	1.05	.066	.988
.10	1.00	.063	1.012
.20	0.70	.038	1.663
.30	.52	.030	1.856
.40	.40	.026	1.952
.50	.30	.024	2.00
1-Cyclohexyltetrazole			
0.0	...	0.107	
.01	2.00	.104	0.07
.02	1.70	.097	.28
.03	1.52	.092	.41
.04	1.40	.087	.53
.05	1.30	.082	.66
.06	1.22	.079	.74
.07	1.15	.077	.79
.08	1.10	.073	.89
.09	1.05	.071	.94
.10	1.00	.066	1.07
.20	0.70	.048	1.53
.25	.60	.041	1.71
.40	.40	.033	1.92
.45	.35	.038	1.79
.50	.30	.030	2.00

The stability constants for the species ML_n, where M = metal and L = ligand, is defined by the expression[14]

$$\beta_n = [ML_n]/[M][L]^n$$

For the case of two complexes one can write

$$\bar{n}(1 - \bar{n})[L] = \beta_1 + \beta_2 \frac{(2 - \bar{n})[L]}{1 - \bar{n}} \qquad (1)$$

Thus knowledge of $\bar{n}$ and [L] allows determination of β_1 and β_2 from a plot of $\bar{n}/(1 - \bar{n})[L]$ *vs.* $(2 - \bar{n})[L]/(1 - \bar{n})$; the slope yields β_2 and the intercept β_1, which were determined by least squares and gave the values for the formation constants shown in Table V.

1-Methyl- and 1-cyclohexyltetrazole complexes of each metal are of comparable strength. It is unfortunate that the same solvent could not be employed for both cobalt and nickel, so direct comparison could be made. Beer's law is not obeyed for $CoCl_2 \cdot 6H_2O$ in ethanol and $NiCl_2$ is insufficiently soluble in tetrahydrofuran.

The formation of the 1-phenyltetrazole nickel complex was studied in tetrahydrofuran. $\beta_1 \approx 4$ and $\beta_2 \approx 3.5 \times 10^3$ after all points more than three times the standard deviation from the "least squares" fit of eq. 1 were rejected. However most of the data between $\bar{n} = 0.5$ and $\bar{n} = 1.3$ were thus rejected and the shape of the formation curve above $\bar{n} = 1.3$ is abnormal, so that we feel the results may not be significant.

[14] F. Rossotti and H. Rossotti, *Acta Chem. Scand.*, **9**, 1166 (1955).

TABLE IV
ABSORBANCIES AND $\bar{n}$ VALUES FOR 1-METHYL- AND 1-CYCOOHEXYLTETRAZOLE AND 0.001 M COBALT CHLORIDE HEXAHYDRATE IN TETRAHYDROFURAN

[L], M	−log [L]	A605 mμ	$\bar{n}$
	1-Methyltetrazole		
0.000		0.108	0.000
.003	2.52	.138	.316
.004	2.40	.145	.388
.005	2.30	.153	.469
.006	2.22	.159	.530
.007	2.15	.165	.592
.008	2.10	.171	.653
.009	2.05	.174	.683
.010	2.00	.180	.745
.030	1.52	.230	1.255
.050	1.30	.247	1.428
.500	0.30	.305	2.000
	1-Cyclohexyltetrazole		
0.000		0.108	0.000
.010	2.00	.185	.773
.012	1.92	.190	.829
.014	1.86	.198	.917
.016	1.80	.202	.961
.018	1.74	.209	1.028
.020	1.70	.209	1.028
.040	1.40	.238	1.359
.060	1.22	.265	1.657
.080	1.10	.278	1.801
.100	1.00	.290	1.934
.500	0.30	.318	2.000

TABLE V
FORMATION CONSTANTS FOR SOME COMPLEXES OF COBALT AND NICKEL WITH 1-SUBSTITUTED TETRAZOLES AT 25°; 90% CONFIDENCE LIMITS ARE GIVEN

Metal halide	Tetrazole	β_1	β_2	Solvent
$NiCl_2$	1-Methyl	3.8 ± 3.8	112 ± 10	Absolute ethanol
$NiCl_2$	1-Cyclohexyl	13.8 ± 4.0	112 ± 12	Absolute ethanol
$CoCl_2$	1-Methyl	135 ± 5	$3.3 \pm 0.1 \times 10^3$	Tetrahydrofuran
$CoCl_2$	1-Cyclohexyl	145 ± 25	$3.2 \pm 0.5 \times 10^3$	Tetrahydrofuran

Infrared spectra show no uniform shifts which can be associated with the coordination to the metals. The hydrochlorides, wherein bonding of the additional proton is probably at the 1-nitrogen, cause the "ring" vibrations[12] to shift to lower frequencies, while the shifts occur toward higher frequencies for the metal complexes, thus supporting the idea that there is a general metal–ring interaction.

At present it is difficult to speculate concerning the nature or site of the metal–tetrazole link. If tetrazoles were satisfying two coordination sites, then one might expect the solids would be ionic, $MT_2^{+2} + 2Cl^-$, and so have solubility in polar solvents. If tetrazole satisfied only one site, then a neutral species, MT_2Cl_2, would possibly result and greater solubility in nonpolar solvents would be expected. It seems likely the polymeric species may have formed.[2] In solution the species are probably simple monomeric ions or molecules, but upon crystallization polymeric species may form.

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CONTRIBUTION FROM THE COATES CHEMICAL LABORATORIES,
LOUISIANA STATE UNIVERSITY, BATON ROUGE, LOUISIANA

Studies on Some Metal Chelates of β-Furfuraldoxime[1a]

BY KANAN M. J. AL-KOMSER[1b,c] AND BUDDHADEV SEN[1d]

Received April 22, 1963

Metal chelate formation by β-furfuraldoxime, a weak acid, was followed potentiometrically by observing the continuous change in hydrogen ion activity using Bjerrum's titration technique. The titrations were performed in a mixed solvent of 75% dioxane and 25% water by volume in an atmosphere of nitrogen. The step formation constants of metal β-furfuraldoximates at different ionic strengths and temperatures were computed through the determination of degree of formation of the system, $\bar{n}$, and measurement of unbound ligand concentration. The metals studied were chromium(III), iron(III), cobalt(III), nickel(II), copper(II), zinc, and lead. The order of magnitude of the stability constants and other indirect evidences indicated that the anion of β-furfuraldoxime exerted a strong ligand field. The values of the formation constants in mixed solvent were extrapolated to zero ionic strength, and these have been defined as the thermodynamic formation constants. The difference between this constant and aqueous standard constant has been explained. Standard entropy change for chelation in the case of iron(III) was a large negative quantity.

We have undertaken a comprehensive study of a number of chelates involving related ligands with a long-range view to correlate the thermodynamics of chelation, absorption spectra, magnetic properties, and thermal stability of these chelates with respect to solvent, properties of the central atom (ionization potential, d-orbital configuration, oxidation state), properties of donor atoms, and their steric orientation in a ligand molecule. This paper reports the thermodynamics of chelation by β-furfuraldoxime. Although it appears that of the various aspects of a chelate men-

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