

前期第一・第二・第三系列主遷移金属-EDTA キレート の構造比較：六座・6配位は主要化学種か？

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Structural comparison of early first-, second- and third-row main-transition metal-EDTA chelates: Is hexadentately six-coordinated species major?

by

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Crystal structures of early first-row, second-row, and third-row main-transition metal-complexes chelated by ethylenediaminetetraacetic acid (EDTA) are surveyed to clarify whether the well-known hexadentately six-coordinate (6&6) species is major or not. In the early first-row transition metal (Sc, Ti, V, Cr, and Mn)-EDTA, only three of 6&6 species were found within 28 kinds. Whereas there were no such 6&6 species within 50 kinds of the second-row, 15 kinds of the third-row main-transition metal-EDTA. It has been also shown that in the late first-row, there were 31 cases of 6&6 species within 99 kinds by previous study. Therefore, the well-known hexadentately six-coordinate structure is not so much major as minor in the main-transition metal-EDTA complexes from the structural comparison.

Key words: Complex ion, Coordination number, D-block elements, Denticity, X-ray crystallography.

1. はじめに

Stezowski & Hoard (1984) [1]は、1983年までに報告されケンブリッジ結晶学データセンター (CCDC) にデータが登録された 46 種類の重金属-EDTA キレートの結晶構造データをまとめた。以降、金属-EDTA キレートの結晶構造データが増え続ける中で、Pb-EDTA (Davidovich 2005) [2]や Bi-EDTA (Stavila et al. 2006) [3]のように特定の金属-EDTA キレートの結晶構造を総説した文献も見られるが、まとめられてない EDTA キレートの結晶構造も多い。そこで筆者は既報の結晶構造解析研究に基づき、後期第一系列遷移金属-EDTA 錯体の配位数と配位座数をまとめた(野口 2023) [4]。本研究報告では前期第一系列・第二系列および第三系列主遷移金属-EDTA 錯体の結晶構造データを報告した文献に基づき、それらの配位数と配位座数を総覧する。そして分析化学、特にキレート滴定法で典型的に示されることが多い 6 配位・六座構造を有している金属-EDTA 錯体と、そうでない配位数や配位座数のもの

が、遷移金属-EDTA キレートにおいてどれだけ知られているかを、先行研究を踏まえつつ明らかにする。

2. 結果と考察

表 1 に、結晶構造データが報告されている前期第一系列遷移金属-EDTA キレート (Sc-EDTA, Ti-EDTA, V-EDTA, Cr-EDTA, Mn-EDTA) について、化学式、配位数、配位座数および文献をまとめた。同様に、表 2 に第二系列遷移金属-EDTA キレート (Y-EDTA, Zr-EDTA, Nb-EDTA, Mo-EDTA, Tc-EDTA, Ru-EDTA, Rh-EDTA, Pd-EDTA, Cd-EDTA)、および第三系列主遷移金属-EDTA キレート (Hf-EDTA, W-EDTA, Re-EDTA, Os-EDTA, Ir-EDTA, Pt-EDTA, Hg-EDTA) を示した。例えば [Nb(EDTA-4H)(H₂O)₂]·2H₂O (Ooi et al. 1998) [41]のように、CCDC に登録された cif ファイルが正常に開けないものもあった。

第三系列主遷移金属-EDTA において、Ta-EDTA は Nb-EDTA と同形であるという (Vuletic et al. 1979) [95]。

表 1 前期第一系列遷移金属-EDTA キレート の化学式, 配位数, 配位座数, 文献

化学式	配位数	配位座数	文献	化学式	配位数	配位座数	文献
(1) Sc-EDTA 錯体				$\text{NH}_4[\text{Sc}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})_2] \cdot 3\text{H}_2\text{O}$	8	6	[5]
(2) Ti-EDTA 錯体							
$[\text{Ti}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})]$	7	6	[6,7]	$\text{Na}_2[\text{Ti}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})_2] \cdot \text{NaCl} \cdot 7\text{H}_2\text{O}$	7	6	[9]
$\text{Na}[\text{Ti}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})] \cdot 2\text{H}_2\text{O}$	7	6	[8]	$\text{K}[\text{Ti}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})] \cdot 2.5\text{H}_2\text{O}$	7	6	
$\text{Ba}[\text{Ti}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})]\text{Cl} \cdot 6\text{H}_2\text{O}$	7	6	[9]	$[\text{Ti}(\text{EDTA}-3\text{H})(\text{H}_2\text{O})] \cdot \text{H}_2\text{O}$	7	6	[10]
(3) V-EDTA 錯体							
$\text{NH}_4[\text{VO}_2(\text{EDTA}-2\text{H})] \cdot 3\text{H}_2\text{O}$	6	4	[11]	$\text{NH}_4[\text{V}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})] \cdot 2.5\text{H}_2\text{O}$	7	6	[17]
$\text{Na}_3[\text{VO}_2(\text{EDTA}-4\text{H})] \cdot 4\text{H}_2\text{O}$	6	4	[12]	$(\text{NH}_4)_3[\text{VO}(\text{O}_2)(\text{EDTA}-3\text{H})]_2$	7	4	[18]
$\text{Ba}[\text{VO}(\text{EDTA}-4\text{H})] \cdot 6\text{H}_2\text{O}$	6	5	[13,14]	$\text{K}_3[\text{VO}(\text{O}_2)(\text{EDTA}-3\text{H})]_2 \cdot 1.6\text{H}_2\text{O}$	7	4	
$\text{Na}[\text{VO}(\text{EDTA}-3\text{H})] \cdot 4\text{H}_2\text{O}$	6	5	[15]	$[\text{V}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})] \cdot \text{H}_2\text{O}$	7	6	[10]
$\text{Na}[\text{V}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})] \cdot 3\text{H}_2\text{O}$	7	6	[16,17]	$[\text{Mg}(\text{H}_2\text{O})_6][\text{VO}(\text{EDTA}-4\text{H})] \cdot 3.5\text{H}_2\text{O}$	6	5	[19]
(4) Cr-EDTA 錯体							
$[\text{Cr}(\text{EDTA}-3\text{H})(\text{H}_2\text{O})]$	6	5	[20-22]	$[\text{Cu}(\text{Tsc})_2][\text{Cr}(\text{EDTA}-4\text{H})]_2$	6	6	[23]
$[\text{Ni}(\text{Tsc})_2][\text{Cr}(\text{EDTA}-4\text{H})]_2$	6	6	[23]	Tsc= thiosemicarbazide, Phen = 1,10-phenanthroline			
(5) Mn-EDTA 錯体							
$\text{Mn}_3(\text{EDTA}-3\text{H})_2 \cdot 10\text{H}_2\text{O}$	7	6	[24-27]	$[\text{Mn}_2(\text{EDTA}-4\text{H})(\text{Phen})] \cdot 4\text{H}_2\text{O}$	7	6	[32]
$\text{Li}_2[\text{Mn}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})] \cdot 4\text{H}_2\text{O}$	7	6	[28]	$(\text{NH}_4)_2[\text{Mn}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})] \cdot 3\text{H}_2\text{O}$	7	6	[33]
$\text{K}[\text{Mn}(\text{EDTA}-3\text{H})] \cdot 2\text{H}_2\text{O}$	6	6	[29,30]	$[\text{Mn}_2(\text{EDTA}-4\text{H})(\text{H}_2\text{O})]_n \cdot n\text{H}_2\text{O}$	7	6	[34]
$\text{Nd}_2\text{Mn}_3(\text{EDTA}-4\text{H})_3(\text{H}_2\text{O})_{11} \cdot 12\text{H}_2\text{O}$	7	6	[31]	$[\text{SrMn}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})_5] \cdot 3/2\text{H}_2\text{O}$	7	6	[35]

表 2 第二系列および第三系列主遷移金属-EDTA キレート の化学式, 配位数, 配位座数, 文献

化学式	配位数	配位座数	文献	化学式	配位数	配位座数	文献
(1) Y-EDTA 錯体							
$\text{NH}_4[\text{Y}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})_3] \cdot 3\text{H}_2\text{O}$	9	6	[36]	$\text{Na}[\text{Y}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})_3] \cdot 5\text{H}_2\text{O}$	9	6	[37]
(2) Zr-EDTA 錯体							
$[\text{Zr}(\text{EDTA}-4\text{H})] \cdot 4\text{H}_2\text{O}$	8	6	[38]	$[\text{ZrO}(\text{EDTA}-4\text{H})]\text{Fe}(\text{OH}) \cdot \text{H}_2\text{O}$	7	6	[40]
$(\text{H}_2\text{N})_2\text{C}=\text{NH}_2[\text{Zr}(\text{EDTA}-4\text{H})\text{CO}_3] \cdot 3\text{H}_2\text{O}$	8	6	[39]				
(3) Nb-EDTA 錯体				$[\text{Nb}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$	8	6	[41]
(4) Mo-EDTA 錯体							
<i>anti</i> - $\text{Na}_4[\text{Mo}_2\text{O}_4(\text{EDTA}-4\text{H})] \cdot 4\text{H}_2\text{O}$	6×2	3	[42]	<i>anti</i> - $\text{Na}_2\text{K}_2[\text{Mo}_2\text{O}_6(\text{EDTA}-4\text{H})] \cdot 10\text{H}_2\text{O}$	6×2	3	[52]
<i>syn</i> - $\text{Cs}_2[\text{Mo}_2\text{S}_2\text{O}_2(\text{EDTA}-4\text{H})] \cdot 2\text{H}_2\text{O}$	7×2	(6)	[43]	<i>syn</i> - $[\text{Gd}(\text{H}_2\text{O})\text{MoO}_4]_2 \cdot [\text{Mo}_2\text{O}_4(\text{EDTA}-4\text{H})]$	7×2	(6)	[53]
<i>syn</i> - $\text{K}[\text{Mo}_2(\text{OH})_2(\text{EDTA}-4\text{H})(\text{CH}_3\text{COO})]$	7×2	(6)	[44]	<i>syn</i> - $[\text{Eu}(\text{H}_2\text{O})\text{MoO}_4]_2 \cdot [\text{Mo}_2\text{O}_4(\text{EDTA}-4\text{H})]$	7×2	(6)	
<i>syn</i> - $\text{K}_4[\text{Mo}_2\text{O}_4(\text{OH})_2(\text{EDTA}-4\text{H})] \cdot 16.4\text{H}_2\text{O}$	7×2	(6)	[45]	<i>anti</i> - $\text{Na}_8[\text{Mo}_{10}\text{O}_{32}(\text{EDTA}-4\text{H})(\text{H}_2\text{O})_{35}]$	6×2	3	[54]
<i>anti</i> - $\text{Na}_4[\text{Mo}_6\text{O}_8(\text{EDTA}-4\text{H})_3] \cdot 14\text{H}_2\text{O}$	8×2	3	[46]	<i>anti</i> - $\text{Na}_2(\text{H}_2\text{O})_6[\text{Mo}_2\text{O}_6(\text{EDTA}-2\text{H})] \cdot 2\text{H}_2\text{O}$	6×2	3	
<i>syn</i> - $\text{Na}_2[\text{Mo}_2\text{O}_4(\text{EDTA}-4\text{H})] \cdot 5\text{H}_2\text{O}$	7×2	(6)	[47]	<i>anti</i> - $\text{Na}_4(\text{H}_2\text{O})_8[\text{Mo}_2\text{O}_6(\text{EDTA}-4\text{H})]$	6×2	3	[55]
<i>syn</i> - $\text{Na}_2[\text{MoWO}_4(\text{EDTA}-4\text{H})] \cdot 4.5\text{H}_2\text{O}$	7,(7)	(6)	[48]	<i>anti</i> - $\text{H}_2\text{temed}[\text{Mo}_2\text{O}_6(\text{EDTA}-2\text{H})] \cdot \text{H}_2\text{O}$	6×2	3	
<i>syn</i> - $\text{Na}_2[\text{Mo}_2\text{O}_4(\text{EDTA}-4\text{H})] \cdot 4\text{H}_2\text{O}$	7×2	(6)	[49]	<i>syn</i> - $\text{H}_2\text{temed}[\text{Mo}_2\text{O}_4\text{EDTA}-4\text{H}] \cdot \text{H}_2\text{O}$	6×2	(6)	[56]
<i>syn</i> - $\text{Ca}[\text{Mo}_2\text{O}_4(\text{EDTA}-4\text{H})] \cdot 9\text{H}_2\text{O}$	7×2	(6)	[50]	<i>anti</i> - $(\text{H}_3\text{NCH}_2\text{CH}_2)_2 \cdot [\text{Mo}_2\text{O}_6(\text{EDTA}-2\text{H})]$	6×2	3	
<i>syn</i> - $\text{Na}_2[\text{Mo}_2\text{O}_5\text{S}(\text{EDTA}-4\text{H})] \cdot 2\text{H}_2\text{O}$	6×2	(6)	[51]	temed=tetramethylethylenediamine			
(5) Tc-EDTA 錯体							
$[\text{Tc}_2\text{O}_2(\text{EDTA}-2\text{H})_2] \cdot 5\text{H}_2\text{O}$	7×2	4	[57]	$\{\text{TcN}((\text{NH}_2)_2\text{CS})_4(\text{EDTA}-4\text{H})\}_2 \cdot 6\text{H}_2\text{O}$		6	3
$\text{Ba}[\text{TcO}(\text{EDTA}-4\text{H})]_2$	7	6	[58]				

(6) Ru-EDTA 錯体							
[Ru(EDTA-3H)(H ₂ O)]	6	5	[60-62]	[Ru(PPh ₃)(EDTA-3H)]·1.5H ₂ O	6	5	[68]
[Ru(EDTA-3H)NO]	6	5	[62-64]	NH ₄ [Ru(EDTA-3H)Cl]·2H ₂ O	6	5	[69]
[Ru(EDTA-2H)(dppm)]·DMSO·H ₂ O	6	4	[65]	[Ru(EDTA-2H)(C ₆ H ₅ S) ₂]	7	4	[70]
K[Ru(EDTA-2H)Cl ₂]	6	4	[66]	[Ru(EDTA-2H)(2-OMe-PhCONHO)]	6	4	[71]
K[Ru(EDTA-3H)Cl]·2H ₂ O	6	5	[67]				
(7) Rh-EDTA 錯体				[Rh(EDTA-3H)(H ₂ O)]	6	5	[72]
(8) Pd-EDTA 錯体							
[PdCl ₂ (EDTA)]·5H ₂ O	4	2	[73]	[PdCl ₂ (EDTA)]·6H ₂ O	4	2	[75]
Ba ₂ [Pd ₂ (EDTA-4H) ₂]·10H ₂ O	4×2	2	[74]	[Pd ₂ (EDTA-2H)] ₂ ·2NaNO ₃ ·7.5H ₂ O	4×2	2	[76]
(9) Cd-EDTA 錯体							
[Mg(H ₂ O) ₆][Cd(EDTA-4H)(H ₂ O)]·3H ₂ O	7	6	[77]	Na ₂ [Cd(H ₂ O) ₄][Cd(EDTA-4H)(H ₂ O)] ₂ ·2H ₂ O	7	6	[80]
[Mn(H ₂ O) ₄][Cd(EDTA-4H)]·2H ₂ O	7	6	[78]	K ₂ [Cd(H ₂ O) ₄][Cd(EDTA-4H)(H ₂ O)] ₂ ·2H ₂ O	7	6	
[Cd{Cd(EDTA-4H)(H ₂ O)} ₂ (H ₂ O) ₄]·4H ₂ O	7	6	[79]	{[Cd ₂ (EDTA-4H)(H ₂ O)]·H ₂ O} _n	7	6	[81]
[Cd(EDTA-2H)(H ₂ O)]·2H ₂ O	7	6	[27]	N ₂ H ₅ [Cd(EDTA-3H)(H ₂ O)]·H ₂ O	7	6	[82]
(10) Hf-EDTA 錯体				(CN ₅ H ₆) ₂ [Hf(EDTA-4H)F ₂]·H ₂ O	8	6	[83]
(11) W-EDTA 錯体							
<i>syn</i> -Ba[W ₂ O ₆ (EDTA-4H)]·3.5H ₂ O	7×2	(6)	[84]	<i>syn</i> -Na ₂ [W ₂ O ₆ S ₂ (EDTA-4H)]·6H ₂ O	7×2	(6)	[86]
<i>syn</i> -Na ₂ [MoWO ₆ (EDTA-4H)]·4.5H ₂ O	7,(7)	(6)	[48]	<i>anti</i> -Na ₄ [W ₂ O ₆ (EDTA-4H)]·8H ₂ O	6×2	3	[52]
<i>syn</i> -NaNH ₄ [W ₂ O ₆ S ₂ (EDTA-4H)]·2H ₂ O	6×2	(6)	[85]	<i>anti</i> -(NH ₄) ₄ [W ₂ O ₆ (EDTA-4H)]·4H ₂ O	6×2	3	[87]
(12) Re-EDTA 錯体				Ba ₂ [Re ₂ O ₇ (EDTA-4H)]·4.5H ₂ O	7	4	[88]
(13) Os-EDTA				[Os(EDTA-4H)(H ₂ O)]·H ₂ O	7	6	[89]
(14) Ir-EDTA							
K[Ir(EDTA-2H)Cl ₂]	6	4	[90]	[(Cp*Ir) ₂ (EDTA-4H)]·4H ₂ O	8×2	3	[91]
K[Ir(EDTA-3H)Cl]·H ₂ O	6	5					
(15) Pt-EDTA				[PtCl ₂ (EDTA)]·6H ₂ O	4	2	[92]
(16) Hg-EDTA							
[Hg(EDTA-2H)]·H ₂ O	6	7	[93]	Li ₄ [Hg ₂ (EDTA-4H) ₂]·8H ₂ O	7	6	[94]

DMSO=dimethylsulfoxide, dppm=bis(diphenylphosphino)methane, Cp*=pentamethylcyclopentadienyl

Ag⁺を含むものは、いずれも EDTA アニオンの内圏で配位されてはいない (Solans et al. 1984 [96], Brouca-Cabarrecq et al. 1996) [97]. また, Au³⁺に第三級アミノ基の窒素原子が配位した錯体の結晶構造はほとんど報告されていない (Cornejo et al. 2003) [98].

前期第一系列遷移金属-EDTA キレートのうち、配位数 6 が 9 種類、配位数 7 が 18 種類、配位数 8 が 1 種類あった。キレート滴定法で学ばれることの多い典型的な六座・6 配位構造のものはわずか 3 種類 (約 11%) にとどまった。後期第一系列遷移金属-EDTA キレートであっても、六座・6 配位構造のものは 99 種類中 31 種類 (約 31%) に過ぎず (野口 2023) [4], 第二系列, 第三系列主遷移金属-EDTA キレートでは、そもそも六座・6 配位構造のものが見られなかった。なお, 希土類金属-EDTA 錯体の配位数は、8 から 10 である (Wang et al. 2010) [99].

以上より、結晶構造が明らかとなっているもの全体

からすると、キレートの代表例として広く知られている金属-EDTA 錯体の六座・6 配位構造は、メジャーというよりはむしろマイナーな化学種である。

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