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Crystal Structures of Ethylenediaminetetraacetato Metal Complexes. VII. Structures containing the $[\text{Fe}(\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_8)(\text{H}_2\text{O})]^-$ Anion: Ethylenediammonium (EDA) and Tetraethylammonium (TEA) Salts

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Abstract. (EDA,Fe): $[\text{C}_2\text{H}_{10}\text{N}_2][\text{Fe}(\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_8)(\text{H}_2\text{O})]_2 \cdot 6\text{H}_2\text{O}$, $M_r = 894.36$, monoclinic, $C2/c$, $a = 30.460$ (3), $b = 7.504$ (1), $c = 15.450$ (2) Å, $\beta = 95.50$ (2)°, $V = 3515$ (1) Å³, $D_x = 1.69$ Mg m⁻³, $Z = 4$, $F(000) = 1872$, $\lambda(\text{Mo } K\alpha) = 0.7093$ Å, $\mu(\text{Mo } K\alpha) = 0.957$ mm⁻¹. (TEA,Fe): $[\text{N}(\text{C}_2\text{H}_5)_4][\text{Fe}(\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_8)(\text{H}_2\text{O})]_2 \cdot 2\text{H}_2\text{O}$, $M_r = 528.36$, orthorhombic, $Pcab$ (non-standard setting of $Pbca$), $a = 22.484$ (4), $b = 15.221$ (3), $c = 14.228$ (3) Å, $V = 4869$ (2) Å³, $D_x = 1.44$ Mg m⁻³, $Z = 8$, $F(000) = 2248$, $\lambda(\text{Mo } K\alpha) = 0.7093$ Å, $\mu(\text{Mo } K\alpha) = 0.700$ mm⁻¹. The structures were refined by full-matrix least squares to R values of 0.041 and 0.065 for 2436 and 2837 observed reflections, respectively. A half-normal probability plot comparison between the $[\text{EDTA}(\text{H}_2\text{O})\text{Fe}]^-$ anions of different structures in the literature shows that the Fe coordination polyhedron in (EDA,Fe) is similar to that in the crystal structure of (Ag,Fe), while the polyhedron in (TEA,Fe) is similar to that in the crystal structure of (K,Fe) [Solans, Font Altaba & Garcia-Oricain (1984). *Acta Cryst.* **C40**, 635–638].

Introduction. A systematic crystal structure study of ethylenediaminetetraacetato complexes is being carried out in the authors' departments (Solans, Font-Altaba & Garcia-Oricain, 1984a,b).

In preceding papers (parts V and VI) the crystal structures of $X[\text{EDTA}(\text{H}_2\text{O})\text{Fe}]_m \cdot n\text{H}_2\text{O}$ with $X = \text{Ag}^+$, Na^+ , K^+ and Tl^+ (Solans *et al.*, 1984a) and Ba^{2+} (Solans *et al.*, 1984b) were determined and compared with the structures where $X = \text{Li}^+$ and Rb^+ (Lind, Hamor, Hamor & Hoard, 1964). Two kinds of $[\text{EDTA}(\text{H}_2\text{O})\text{Fe}]$ bipyramidal-pentagonal coordination polyhedra were found. In this paper the influence of organic cations is studied; two ions have been selected, one with spherical symmetry $[\text{N}(\text{C}_2\text{H}_5)_4]^+$, tetraethylammonium] and the other without this symmetry ($\text{C}_2\text{H}_{10}\text{N}_2^+$, ethylenediammonium).

Experimental. (EDA,Fe): yellow-brown prism $0.1 \times 0.1 \times 0.2$ mm. (TEA,Fe): red prism $0.2 \times 0.2 \times 0.3$ mm. Philips PW 1100 diffractometer, graphite monochromator, Mo $K\alpha$. Cell parameters in both structures from 25 reflections ($4 \leq \theta \leq 12^\circ$). ω -scan technique, scan width 0.8° , scan speed $0.03^\circ \text{ s}^{-1}$. Three standard reflections every 2 h, no variations in reference intensities. (EDA,Fe): 2454 reflections measured; $\theta \leq 30^\circ$; h,k,l : $\pm 36,8,18$; 2436 with $I \geq 2.5\sigma(I)$. (TEA,Fe): 2891 reflections measured; $\theta \leq 30^\circ$; h,k,l : 24,18,16; 2837 with $I \geq 2.5\sigma(I)$. Lp corrections, absorption ignored. (EDA,Fe) determined by direct methods with *MULTAN80* (Main, Fiske, Hull,

Lessinger, Germain, Declercq & Woolfson, 1980). Fe atom of (TEA,Fe) from Patterson map (*SHELX76*, Sheldrick, 1976), remaining non-hydrogen atoms from a weighted Fourier synthesis. Full-matrix least-squares refinement of coordinates and U_{ij} 's (*SHELX76*), $\sum w|F_o| - |F_c|^2$ minimized, $w = [\sigma^2(F_o) + k|F_o|^2]^{-1}$, where $k = 0.00417$ in (EDA,Fe) and 0.0211 in (TEA,Fe); f , f' and f'' from *International Tables for*

X-ray Crystallography (1974). ΔF map shows double peaks for four C atoms of the tetraethylammonium ion in (TEA,Fe), which were refined with an occupancy factor of 0.5, according to heights of peaks. H atoms of EDTA ligand in both structures were calculated and refined with an overall isotropic temperature factor and constrained bond length. 10 H in (EDA,Fe) from ΔF synthesis and refined with the same overall isotropic

Table 1. Atomic coordinates ($\times 10^4$, Fe $\times 10^5$) and equivalent isotropic temperature factors (\AA^2)

$$B_{eq} = \frac{1}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B_{eq}
(EDA,Fe)				
Fe	15156 (2)	29761 (7)	31172 (3)	1.44 (2)
N(10)	908 (1)	2969 (4)	3914 (2)	1.7 (1)
C(10)	533 (1)	2002 (5)	3435 (3)	2.1 (2)
C(20)	704 (1)	245 (5)	3117 (3)	2.0 (2)
N(20)	1079 (1)	563 (4)	2593 (2)	1.7 (1)
O(11)	1558 (1)	5215 (4)	3894 (2)	2.2 (1)
C(11)	1229 (1)	5871 (5)	4250 (2)	2.0 (2)
O(12)	1246 (1)	7238 (4)	4698 (2)	2.8 (1)
C(12)	801 (1)	4871 (5)	4049 (3)	2.1 (2)
O(21)	1729 (1)	1473 (3)	4129 (2)	2.0 (1)
C(21)	1522 (1)	1388 (5)	4827 (2)	1.8 (2)
O(22)	1677 (1)	762 (4)	5514 (2)	2.6 (1)
C(22)	1054 (1)	2117 (6)	4760 (3)	2.4 (2)
O(31)	1121 (1)	4089 (3)	2169 (2)	2.2 (1)
C(31)	860 (1)	3138 (5)	1644 (2)	2.0 (2)
O(32)	572 (1)	3779 (4)	1120 (2)	2.9 (1)
C(32)	924 (1)	1131 (5)	1693 (3)	2.0 (2)
O(41)	1910 (1)	1224 (3)	2439 (2)	1.7 (1)
C(41)	1835 (1)	-473 (5)	2478 (2)	1.6 (2)
O(42)	2121 (1)	-1638 (3)	2453 (2)	2.3 (1)
C(42)	1370 (1)	-1030 (5)	2597 (3)	1.9 (2)
N	2072 (1)	2836 (5)	780 (2)	2.4 (2)
C	2523 (2)	2869 (6)	-446 (3)	2.5 (2)
O(W1)	2039 (1)	4519 (4)	2680 (2)	2.6 (1)
O(W2)	4779 (1)	1994 (5)	3237 (3)	4.9 (2)
O(W3)	4830 (1)	3463 (5)	275 (2)	4.9 (2)
O(W4)	2326 (1)	3664 (4)	5804 (3)	3.5 (2)
(TEA,Fe)				
Fe	14736 (3)	29400 (5)	8800 (5)	2.07 (3)
N(10)	501 (2)	3393 (3)	840 (3)	2.7 (2)
C(10)	197 (3)	3246 (5)	1755 (4)	3.2 (3)
C(20)	617 (3)	3438 (4)	2551 (4)	2.9 (2)
N(20)	1142 (2)	2864 (3)	2452 (3)	2.2 (2)
O(11)	1182 (2)	2867 (3)	-516 (3)	3.5 (2)
C(11)	638 (3)	2785 (5)	-733 (4)	3.5 (3)
O(12)	447 (2)	2592 (5)	-1512 (4)	5.6 (2)
C(12)	206 (3)	2932 (5)	70 (5)	3.6 (3)
O(21)	1605 (2)	4106 (3)	963 (3)	3.2 (2)
C(21)	1201 (3)	4665 (4)	724 (5)	3.5 (3)
O(22)	1305 (3)	5431 (3)	575 (5)	6.0 (3)
C(22)	579 (3)	4329 (5)	628 (5)	3.8 (3)
O(31)	1116 (2)	1680 (3)	1034 (3)	3.4 (2)
C(31)	950 (3)	1365 (4)	1823 (5)	3.6 (3)
O(32)	761 (3)	632 (4)	1915 (4)	6.8 (3)
C(32)	1004 (3)	1948 (4)	2673 (4)	3.0 (3)
O(41)	2200 (2)	2612 (3)	1748 (3)	3.2 (2)
C(41)	2215 (3)	2850 (4)	2607 (4)	2.8 (3)
O(42)	2668 (2)	2830 (4)	3110 (3)	4.2 (2)
C(42)	1643 (3)	3193 (5)	3019 (4)	3.0 (3)
O(W1)	2208 (2)	2571 (3)	8 (3)	3.6 (2)
O(W2)	1311 (2)	2255 (3)	-2845 (3)	3.8 (2)
O(W3)	3376 (2)	2039 (3)	260 (4)	4.4 (3)
N	3454 (2)	4889 (3)	1233 (4)	3.3 (2)
C(51)	3165 (7)	5706 (8)	1510 (11)	4.3 (7)
C(52)	2601 (5)	5528 (9)	2225 (10)	9.8 (7)
C(53)	3646 (8)	4328 (10)	2021 (11)	4.7 (7)
C(54)	4091 (6)	4703 (8)	2732 (9)	8.9 (7)
C(55)	4011 (8)	5188 (11)	635 (12)	5.4 (8)
C(56)	4328 (6)	4284 (11)	231 (11)	11.4 (8)
C(57)	3047 (5)	4344 (8)	593 (9)	2.9 (6)
C(58)	2814 (5)	4999 (12)	-237 (8)	11.1 (9)
C(51')	1405 (11)	4025 (13)	5777 (15)	7.4 (11)
C(53')	1659 (8)	5572 (11)	5544 (12)	5.0 (7)
C(55')	2861 (9)	4691 (14)	1815 (16)	7.7 (11)
C(57')	3939 (9)	5258 (12)	1932 (17)	6.9 (11)

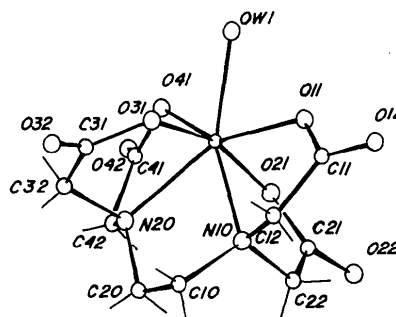


Fig. 1. View of the Fe coordination polyhedron with the numbering of the atoms.

Table 2. Principal bond distances (\AA) and angles ($^\circ$)

	(EDA,Fe)	(TEA,Fe)
Fe-N(10)	2.319 (3)	2.345 (5)
Fe-N(20)	2.345 (3)	2.358 (5)
Fe-O(11)	2.062 (2)	2.092 (4)
Fe-O(41)	2.123 (2)	2.077 (4)
Fe-O(21)	1.986 (2)	1.953 (4)
Fe-O(31)	1.988 (2)	1.954 (4)
Fe-O(W1)	2.132 (2)	2.107 (4)
N(20)-Fe-N(10)	74.1 (1)	73.9 (2)
O(11)-Fe-N(10)	72.6 (1)	71.2 (2)
O(41)-Fe-N(20)	71.3 (1)	71.8 (2)
O(21)-Fe-N(10)	78.3 (1)	77.8 (2)
O(31)-Fe-N(20)	77.7 (1)	77.1 (2)
O(21)-Fe-N(20)	88.0 (1)	88.6 (2)
O(31)-Fe-N(10)	86.3 (1)	86.7 (2)
O(21)-Fe-O(11)	90.4 (1)	94.9 (2)
O(31)-Fe-O(11)	94.9 (1)	89.7 (2)
O(21)-Fe-O(41)	83.1 (1)	90.5 (2)
O(31)-Fe-O(41)	103.2 (1)	96.1 (2)
O(11)-Fe-O(W1)	74.7 (1)	71.9 (2)
O(41)-Fe-O(W1)	72.8 (1)	72.6 (2)
O(21)-Fe-O(W1)	111.1 (1)	96.2 (2)
O(31)-Fe-O(W1)	87.5 (1)	102.3 (2)
N...O(12 ⁱ)	2.885 (4)	
N...O(41 ⁱⁱ)	2.919 (4)	
N...O(W4 ⁱⁱⁱ)	2.738 (4)	
O(W1)...O(42 ⁱⁱⁱ)	2.728 (4)	
O(W2)...O(W2 ^{iv})	2.756 (4)	
O(W3)...O(W2 ^{iv})	2.720 (4)	
O(W3)...O(W3 ^v)	2.698 (4)	
O(W1)...O(42 ⁱ)		2.741 (6)
O(W1)...O(W3 ^{iv})		2.769 (6)
O(W2)...O(12 ⁱⁱ)		2.762 (6)
O(W2)...O(42 ⁱ)		2.809 (6)
O(W3)...O(22 ⁱⁱ)		2.815 (6)
O(W3)...O(W2 ^{iv})		2.806 (6)

Symmetry code

	(EDA,Fe)	(TEA,Fe)
(i)	$x, 1-y, z-\frac{1}{2}$	$\frac{1}{2}-x, y, z-\frac{1}{2}$
(ii)	x, y, z	x, y, z
(iii)	$\frac{1}{2}-x, y+\frac{1}{2}, \frac{1}{2}-z$	$\frac{1}{2}-x, y-\frac{1}{2}, -z$
(iv)	$1-x, y, \frac{1}{2}-z$	$\frac{1}{2}-x, y, \frac{1}{2}+z$
(v)	$1-x, 1-y, -z$	

temperature factor. Final R (wR) 0.041 (0.047) and 0.065 (0.075) for (EDA,Fe) and (TEA,Fe) respectively. $(\Delta/\sigma)_{\max} = 2.6$ in x coordinate of H(O W 1) and 2.7 in U_{13} of C(58), respectively. [These values in the anion are: 1.1 in U_{22} of O(21) and 0.4 in x of O(22), respectively.] Max. and min. peak heights in final ΔF synthesis $\pm 0.3 e \text{ \AA}^{-3}$ in both structures. IBM-4341 computer.

Discussion. Final atomic parameters are in Table 1.*

The crystal structures of (EDA,Fe) and (Ba,Fe) (Solans *et al.*, 1984b) have the same space group and similar cell parameters; the main difference between the two structures is the location of the cation, which lies on an inversion centre in (EDA,Fe), while it is on a twofold axis in (Ba,Fe).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters and a full tabulation of bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39944 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

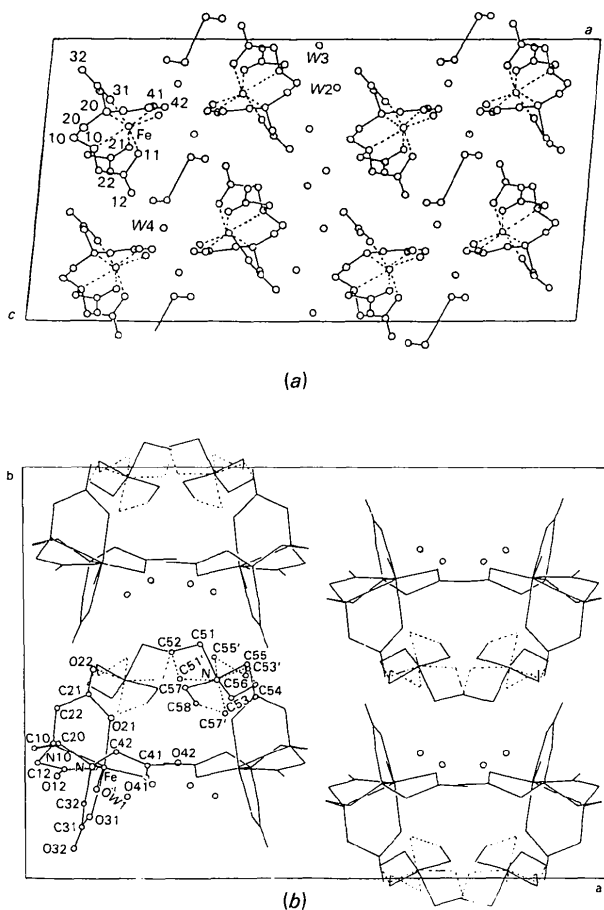


Fig. 2. Views of the unit-cell contents: (a) (EDA,Fe), (b) (TEA,Fe).

Table 3. Results of half-normal probability plot comparison, and average of principal bond angles ($^\circ$) for each type of Fe coordination polyhedron [(I) = (Ag,Fe), (II) = (Li,Fe) and (III) = (K,Fe)]

Comparison	Intercept	Slope	s^2
(Ag,Fe)-(EDA,Fe)	-0.48 (20)	3.55 (20)	0.345
(Ag,Fe)-(K,Fe)	-2.02 (57)	8.98 (58)	2.668
(Ag,Fe)-(Li,Fe)	-3.57 (74)	13.24 (75)	4.441
(Ag,Fe)-(Na,Fe)	-0.23 (5)	1.62 (5)	0.021
(Ag,Fe)-(Rb,Fe)	-0.34 (14)	2.95 (14)	0.145
(Ag,Fe)-(TEA,Fe)	-2.81 (61)	10.22 (61)	3.132
(Ag,Fe)-(Tl,Fe)	-1.85 (46)	8.63 (46)	1.693
(EDA,Fe)-(K,Fe)	-5.8 (16)	20.2 (16)	19.988
(EDA,Fe)-(Li,Fe)	-9.5 (23)	29.2 (23)	42.179
(EDA,Fe)-(Na,Fe)	-0.60 (26)	3.91 (7)	0.593
(EDA,Fe)-(Rb,Fe)	0.41 (9)	3.57 (9)	0.064
(EDA,Fe)-(TEA,Fe)	-5.3 (14)	19.5 (14)	17.343
(EDA,Fe)-(Tl,Fe)	-3.72 (9)	14.40 (9)	6.789
(K,Fe)-(Li,Fe)	-2.04 (66)	8.91 (67)	3.518
(K,Fe)-(Na,Fe)	-2.19 (80)	10.55 (81)	5.245
(K,Fe)-(Rb,Fe)	-2.08 (68)	10.91 (69)	3.806
(K,Fe)-(TEA,Fe)	0.18 (21)	6.23 (21)	0.361
(K,Fe)-(Tl,Fe)	-0.63 (25)	4.28 (25)	0.501
(Li,Fe)-(Na,Fe)	-4.0 (11)	15.1 (11)	10.365
(Li,Fe)-(Rb,Fe)	-4.2 (11)	15.9 (11)	9.348
(Li,Fe)-(TEA,Fe)	-1.17 (23)	4.98 (23)	11.374
(Li,Fe)-(Tl,Fe)	-0.13 (13)	-0.13 (13)	17.134
(Na,Fe)-(Rb,Fe)	-0.32 (12)	2.83 (12)	0.125
(Na,Fe)-(TEA,Fe)	-2.68 (70)	11.80 (71)	4.188
(Na,Fe)-(Tl,Fe)	-1.77 (65)	9.58 (66)	3.435
(Rb,Fe)-(TEA,Fe)	-3.16 (74)	12.65 (75)	4.673
(Rb,Fe)-(Tl,Fe)	-1.67 (51)	12.21 (52)	2.113
(TEA,Fe)-(Tl,Fe)	0.03 (17)	3.03 (17)	0.234

Angle	(I)	(II)	(III)
O(21)-Fe-O(11)	89.2 (12)	91.2	95.2 (11)
O(21)-Fe-O(41)	83.5 (7)	91.4	90.2 (1)
O(21)-Fe-O(W1)	109.1 (23)	100.6	98.1 (23)
O(31)-Fe-O(41)	103.6 (6)	95.5	94.4 (10)
O(31)-Fe-O(W1)	88.6 (10)	93.5	98.5 (2)
O(31)-Fe-O(11)	94.8 (10)	90.4	90.7 (16)

As in the previously determined structures, the Fe^{3+} ion is surrounded by four O atoms and two N atoms of an EDTA ligand, which acts in sixidentate fashion, and by a water molecule. The coordination polyhedron is roughly pentagonal bipyramidal (see Fig. 1). The bond distances (Table 2) are similar to those obtained in the previously determined structures. The packing arrangements are shown in Fig. 2.

In order to classify the Fe coordination a half-normal probability plot comparison has been carried out (Abrahams & Keve, 1971). In the analysis all interatomic distances of the Fe coordination polyhedra were compared. According to the residual variance s^2 , and the slope and intercept (Table 3), the Fe coordination polyhedra can be classified into three types: (I) is shown by the (Ag,Fe), (EDA,Fe), (Na,Fe) and (Rb,Fe) crystal structures, (II) by (Li,Fe) and (III) by (K,Fe), (TEA,Fe) and (Tl,Fe). Comparison with (Ba,Fe) is not possible because of the lower accuracy of this structure determination. The main difference is in the position of the apical O atoms (O_R). The different angles for each type of polyhedron are also listed in Table 3. In crystal structures of type (I) the cation is coordinated to water molecules and O atoms of the EDTA ligand, while in structure type (III) the cation coordinates only with O atoms of the EDTA ligand.

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[1-(2-Carboxyphenyl)-3,5-diphenylformazanato](pyridine)palladium(II), [Pd(C₂₀H₁₄N₄O₂)(C₅H₅N)]

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Abstract. $M_r = 527.9$, monoclinic, $P2_1/n$, $a = 10.169$ (1), $b = 19.487$ (3), $c = 11.050$ (1) Å, $\beta = 97.52$ (2)°, $V = 2170.9$ (5) Å³, $Z = 4$, $D_x = 1.61$ g cm⁻³, $\mu(\text{Mo } K\alpha) = 8.74$ cm⁻¹, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $F(000) = 1064$, room temperature, $R = 0.038$ for 4232 reflexions. Pd has an approximately square-planar coordination in which the terminal N atoms of the formazan moiety [Pd–N 1.969 (4) and 1.987 (4) Å], one O atom of the carboxyl group [Pd–O 2.005 (3) Å] and the pyridine N atom [Pd–N 2.081 (5) Å] are involved.

Introduction. Square-planar complexes of transition-metal d^8 ions with the bidentate dibasic 1-(2-hydroxyphenyl)-3,5-diphenylformazan are interesting model compounds for mechanistic studies on ligand-substitution reactions (Balt & Meuldijk, 1981; Balt, Meuldijk & Wismeijer, 1981). The rigidity of the formazan moiety hinders the formation of a five-coordinated intermediate which is usually formed in substitution reactions at square-planar complexes. Until now complexes of only one tridentate formazan have been reported and to investigate other tridentate formazans the title complex has been isolated.

Experimental. 1-(2-Carboxyphenyl)-3,5-diphenylformazan was prepared according to a method described by Wizinger & Biro (1949). 6 mmol of Pd(NO₃)₂·2H₂O dissolved in a minimum amount of water were added slowly to a boiling solution of 5 mmol of the formazan in 100 cm³ 96% ethanol. The reaction mixture was cooled to room temperature and the precipitate was separated by filtration and dissolved in 150 cm³ boiling trichloromethane. To this solution 25 cm³ and 150 cm³ water were added successively. The organic layer containing PDFORM was separated and filtered. 25 cm³ methanol was added to the filtrate and PDFORM crystals were formed by slow evaporation of the solvent in air. The product was dried *in vacuo* over KOH. Yield: 67%. Analysis (%): calculated for C₂₅H₁₉N₅O₂Pd: Pd, 20.16; C, 56.89; H, 3.63; found: Pd, 19.84; C, 55.90; H, 3.64.

D_m not measured. 33 reflexions (2θ 41–56°) used to measure lattice parameters; $2\theta_{\text{max}} = 60^\circ$, $h - 14$ to 14, $k 0$ to 27, $l 0$ to 15; one standard reflexion, no systematic variation; 6297 unique reflexions, 4232 with $I > 2.5\sigma(I)$; Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation; no absorption correction (crystal dimensions 0.10 × 0.10 × 0.15 mm). Structure solved by means of a Patterson minimum function based on the four Pd atoms in the unit cell. Refinement on F proceeded by anisotropic block-diagonal least-squares calculations during the course of which a ΔF synthesis indicated the H atoms,

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