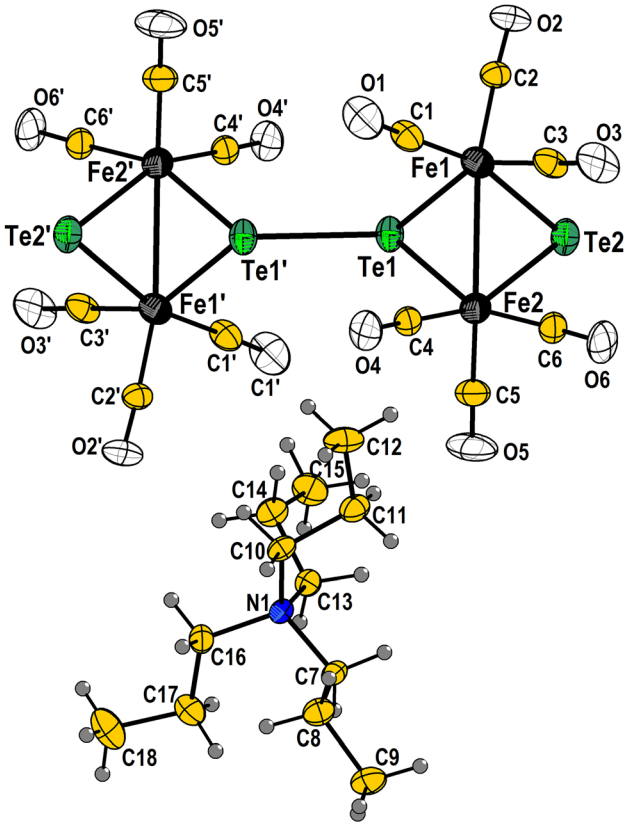
Kang-Woo Kim* and Hyoyi Yang

Crystal structure of bis(tetrapropylammonium) dodecacarbonyltetratelluridotetraferrate(2-), $(Pr_4N)_2[Fe_4Te_4(CO)_{12}]$



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Abstract

$C_{36}H_{56}O_{12}N_2Fe_4Te_4$, triclinic, $P\bar{1}$ (no. 2), a = 11.3615(9) Å, b = 11.8540(9) Å, c = 12.1267(9) Å, α = 61.419(2)°, β = 64.289(3)°, γ = 71.088(3)°, V = 1278.10(17) Å^3, Z = 1, $R_{gt}(F)$ = 0.0213, $wR_{ref}(F^2)$ = 0.0638, T = 223(2) K.

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***Corresponding author: Kang-Woo Kim**, Department of Chemistry & Research Institute for Natural Sciences, Incheon National University, Incheon 22012, Korea, E-mail: kimkw@inu.ac.kr. https://orcid.org/0000-0003-1287-4904

Hyoyi Yang, Department of Chemistry & Research Institute for Natural Sciences, Incheon National University, Incheon 22012, Korea

Table 1: Data collection and handling.

Crystal:	Red polyhedral
Size:	0.26 × 0.19 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	3.40 mm^{-1}
Diffractometer, scan mode:	PHOTON II M14 CPAD, φ and ω
θ_{max}, completeness:	28.3°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	38,939, 6287, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5775
$N(param)_{refined}$:	262
Programs:	Bruker [1], SHELX [2], WinGX/ORTEP [3], Diamond [4]

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

$Fe_3(CO)_{12}$ (0.050 g, 0.10 mmol), Na_2Te_2 (0.030 g, 0.10 mmol) and Pr_4NBr (0.160 g, 0.60 mmol) were charged to a Pyrex tube with diameter of 9 mm under an argon atmosphere and about 0.4 mL MeOH was added as a solvent. While the solvent was being frozen, the Pyrex tube was evacuated under vacuum and sealed with the use of a flame. The sealed tube was placed in an oven and heated at 80 °C for a day, then cooled to room temperature. Dark red polyhedral crystals were isolated by filtration and washed with MeOH and diethyl ether several times. Crystals of $(Pr_4N)_2[Fe_4Te_4(CO)_{12}]$ were obtained in 21 % yield, based on Na_2Te_2.

2 Experimental details

H atoms were positioned geometrically and treated as riding, with C—H = 0.97 (CH_2) and 0.96 (CH_3) Å with U_{iso}(H) = 1.2 (1.5 for methyl) U_{eq}(C). H atoms of the CH_3 were positioned to be staggered with respect to the shortest other bond to the atom to which the CH_3 is attached.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Te1	0.50559 (2)	0.01245 (2)	0.60973 (2)	0.03183 (5)
Te2	0.58570 (2)	−0.06964 (2)	0.85819 (2)	0.03709 (5)
Fe1	0.73278 (3)	−0.08721 (3)	0.63166 (4)	0.03262 (8)
Fe2	0.53223 (4)	−0.21508 (3)	0.78578 (3)	0.03480 (8)
N1	0.1947 (2)	−0.63624 (19)	0.79442 (19)	0.0324 (4)
O1	0.8442 (3)	−0.1441 (4)	0.3948 (3)	0.0920 (10)
O2	0.8767 (2)	0.1233 (2)	0.5411 (3)	0.0650 (7)
O3	0.9009 (3)	−0.2992 (3)	0.7703 (4)	0.0887 (10)
O4	0.5665 (3)	−0.3508 (2)	0.6247 (3)	0.0675 (7)
O5	0.2552 (3)	−0.2154 (4)	0.9540 (4)	0.1016 (12)
O6	0.6457 (4)	−0.4432 (2)	0.9729 (3)	0.0799 (9)
C1	0.7982 (3)	−0.1206 (4)	0.4873 (4)	0.0533 (7)
C2	0.8180 (3)	0.0437 (3)	0.5741 (3)	0.0435 (6)
C3	0.8337 (3)	−0.2157 (3)	0.7161(4)	0.0541 (8)
C4	0.5527 (3)	−0.2958 (3)	0.6860 (3)	0.0439 (6)
C5	0.3634 (4)	−0.2157 (4)	0.8873 (3)	0.0587 (8)
C6	0.6029 (4)	−0.3531 (3)	0.8989 (3)	0.0533 (7)
C7	0.1583 (3)	−0.7480 (2)	0.9296 (2)	0.0369 (5)
H7A	0.214164	−0.756915	0.977051	0.044*
H7B	0.066712	−0.724799	0.980629	0.044*
C8	0.1710 (3)	−0.8794 (3)	0.9295 (3)	0.0469 (6)
H8A	0.264390	−0.913782	0.896230	0.056*
H8B	0.127286	−0.870775	0.871160	0.056*
C9	0.1064 (4)	−0.9712 (3)	1.0720 (4)	0.0617 (9)
H9A	0.113584	−1.055851	1.073853	0.093*
H9B	0.150554	−0.979521	1.128926	0.093*
H9C	0.013927	−0.936664	1.103943	0.093*
C10	0.3378 (2)	−0.6627 (2)	0.7119 (2)	0.0372 (5)
H10A	0.350449	−0.742950	0.700890	0.045*
H10B	0.354797	−0.591991	0.623049	0.045*
C11	0.4389 (3)	−0.6756 (3)	0.7679 (3)	0.0479 (7)
H11A	0.434431	−0.755467	0.848755	0.058*
H11B	0.420027	−0.601889	0.791824	0.058*
C12	0.5763 (3)	−0.6792 (3)	0.6654 (4)	0.0592 (9)
H12A	0.640854	−0.687541	0.702101	0.089*
H12B	0.595026	−0.752830	0.642699	0.089*
H12C	0.580654	−0.599587	0.585891	0.089*
C13	0.1708 (3)	−0.5166 (2)	0.8221 (3)	0.0363 (5)
H13A	0.075468	−0.495025	0.863124	0.044*
H13B	0.212006	−0.539340	0.887634	0.044*
C14	0.2211 (3)	−0.3963 (3)	0.7034 (3)	0.0468 (6)
H14A	0.174431	−0.365950	0.640352	0.056*
H14B	0.315617	−0.416343	0.657602	0.056*
C15	0.1984 (4)	−0.2915 (3)	0.7514 (4)	0.0607 (8)
H15A	0.230455	−0.213828	0.675733	0.091*
H15B	0.104650	−0.271634	0.795962	0.091*
H15C	0.245414	−0.321877	0.813160	0.091*
C16	0.1114 (3)	−0.6148 (3)	0.7144 (3)	0.0426 (6)
H16A	0.127253	−0.533104	0.635815	0.051*
H16B	0.142038	−0.684818	0.682598	0.051*
C17	−0.0358 (3)	−0.6098 (4)	0.7872 (4)	0.0570 (8)
H17A	−0.054171	−0.694243	0.860717	0.068*
H17B	−0.067096	−0.544442	0.825218	0.068*
C18	−0.1091 (5)	−0.5766 (6)	0.6949 (6)	0.0929 (15)
H18A	−0.203070	−0.574036	0.744104	0.139*
H18B	−0.092100	−0.492388	0.622865	0.139*
H18C	−0.079190	−0.642026	0.658325	0.139*

3 Comment

Anionic iron tellurido carbonyl clusters have been under investigation for decades mainly due to their structural novelty and diversity [5–9]. Followings are structurally characterized homometallic anionic iron tellurido carbonyl clusters so far: $[Fe_2Te_3(CO)_6]^{2-}$ [6, 7], $[Fe_3Te(CO)_9]^{2-}$ [8], $[Fe_4Te(CO)_{16}]^{2-}$ [8], $[Fe_4Te_2(CO)_{14}]^{2-}$ [9], $[Fe_5Te_4(CO)_{14}]^{2-}$ [10, 11], $[Fe_6Te_{14}(CO)_{12}]^{2-}$ [7], $[Fe_8Te_6(CO)_{24}]^{2-}$ [12], and $[Fe_8Te_{10}(CO)_{20}]^{2-}$ [10, 13]. Here we report a new compound containing a hitherto unknown homometallic iron tellurido carbonyl cluster, $[Fe_4Te_4(CO)_{12}]^{2-}$. By employing a methanothermal method, $(Pr_4N)_2[Fe_4Te_4(CO)_{12}]$ has been synthesized from the reaction between $Fe_3(CO)_{12}$ and Na_2Te_2 in the presence of Pr_4NBr. There exist Se and S analogue of $[Fe_4Te_4(CO)_{12}]^{2-}$, which is stabilized with Ph_4P^+ and Ph_4As^+ cation, respectively [13, 14].

The $[Fe_4Te_4(CO)_{12}]^{2-}$ anion in $(Pr_4N)_2[Fe_4Te_4(CO)_{12}]$ was revealed to be the Te analogue of $[Fe_4Q_4(CO)_{12}]^{2-}$ (Q = S, Se). The molecular structure of $[Fe_4Te_4(CO)_{12}]^{2-}$ anion can be depicted as $[\{Fe_2Te(CO)_6\}_2(Te_2)]^{2-}$ and considered to be composed of two so called 'butterfly' $Fe_2Te_2(CO)_6$ fragments joining through a Te–Te bond. The two butterfly $Fe_2Te_2(CO)_6$ fragments are crystallographically equivalent by an inversion center located at the midpoint of the ditelluride Te_2^{2-} ligand. There exists an Fe–Fe metal-metal bond in the $Fe_2Te_2(CO)_6$ anion, as the Fe(1)–Fe(2) bond distance is 2.6107(5) Å, which is quite close to the Fe–Fe bond distances found in the other homometallic anionic iron tellurido carbonyl clusters containing the $Fe_2Te_2(CO)_6$ fragment. In $[\{Fe_2Te(CO)_6\}_2(Te_2)]^{2-}$, the Te–Te bond of the ditelluride Te_2^{2-} ligand is slightly longer than the normal Te–Te bonds which typically fall in the range of 2.7–2.8 Å, as the Te(1)–Te(1′) distance is 2.8732(4) Å. Considering the oxidation state of +1 in each butterfly iron atom, charge distribution of the $[Fe_4Te_4(CO)_{12}]^{2-}$ anion can be described as $[(Fe^{+1})_4(Te_2^{-2})(Te^{-2})_2(CO)_{12}]^{2-}$.

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