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Crystal structure of bis(bis(triphenylphosphine)iminium) tetradecacarbonyltetratelluridopentaferate(2-), (PPN)₂[Fe₅Te₄(CO)₁₄]

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Abstract

C₈₆H₆₀O₁₄N₂P₄Fe₅Te₄, triclinic, $P\bar{1}$ (No. 2), $a = 13.6807(7)$ Å, $b = 18.5116(11)$ Å, $c = 19.3210(10)$ Å, $\alpha = 78.170(2)^\circ$, $\beta = 70.1007(18)^\circ$, $\gamma = 70.5341(19)^\circ$, $V = 4314.7(4)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0411$, $wR_{\text{ref}}(F^2) = 0.1234$, $T = 223$ K.

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Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.26 × 0.19 × 0.15 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ :	2.28 mm ⁻¹
Diffractometer, scan mode:	PHOTON II M14 CPAD, φ and ω
θ_{max} , completeness:	28.3°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	145,882, 21,464, 0.046
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 16161
$N(\text{param})_{\text{refined}}$:	1037
Programs:	Bruker [1], SHELX [2], WinGX/ORTEP [3], Diamond [4]

The title crystal 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.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Te1	−0.04632 (2)	1.26067 (2)	0.16784 (2)	0.04128 (8)
Te2	0.13372 (3)	1.12708 (2)	0.22032 (2)	0.05003 (9)
Te3	0.00645 (2)	1.28701 (2)	0.35238 (2)	0.04367 (8)
Te4	−0.23300 (2)	1.29922 (2)	0.36297 (2)	0.04702 (9)
Fe1	−0.05959 (5)	1.19297 (4)	0.30325 (4)	0.04146 (15)
Fe2	0.15816 (5)	1.24057 (4)	0.11915 (4)	0.04608 (16)
Fe3	0.07631 (7)	1.13885 (4)	0.10322 (4)	0.05264 (18)
Fe4	−0.14651 (6)	1.40993 (4)	0.33736 (4)	0.04588 (16)
Fe5	−0.16656 (6)	1.32353 (5)	0.46323 (4)	0.05136 (18)
P1	0.39706 (9)	1.49017 (6)	0.25476 (6)	0.0335 (2)
P2	0.28008 (8)	1.59963 (6)	0.15741 (5)	0.0312 (2)
P3	0.67369 (8)	1.88537 (6)	0.33568 (6)	0.0315 (2)
P4	0.57232 (10)	2.01590 (7)	0.23987 (7)	0.0426 (3)
N1	0.3013 (3)	1.5298 (2)	0.2186 (2)	0.0384 (8)
N2	0.6549 (3)	1.9606 (2)	0.2819 (2)	0.0423 (8)
O1	−0.1901 (4)	1.1123 (3)	0.2756 (2)	0.0695 (11)
O2	−0.0348 (5)	1.0864 (3)	0.4334 (3)	0.0898 (17)
O3	0.1983 (4)	1.3477 (3)	0.1905 (3)	0.0835 (15)
O4	0.1270 (4)	1.3404 (3)	−0.0146 (2)	0.0738 (12)
O5	0.3849 (4)	1.1614 (3)	0.0534 (4)	0.115 (2)
O6	−0.0095 (5)	1.0065 (3)	0.1606 (3)	0.0916 (16)
O7	−0.0186 (7)	1.1979 (4)	−0.0173 (3)	0.133 (3)
O8	0.2875 (6)	1.0576 (6)	0.0109 (6)	0.194 (5)
O9	−0.1081 (4)	1.4609 (3)	0.1798 (2)	0.0818 (14)
O10	−0.3587 (4)	1.5236 (3)	0.3796 (2)	0.0931 (17)
O11	−0.0250 (4)	1.4979 (3)	0.3658 (3)	0.0930 (16)
O12	−0.1642 (5)	1.1839 (3)	0.5655 (3)	0.0938 (17)
O13	−0.3906 (4)	1.4116 (3)	0.5298 (3)	0.0902 (15)
O14	−0.0582 (5)	1.3854 (4)	0.5341 (3)	0.1032 (19)
C1	−0.1344 (4)	1.1428 (3)	0.2844 (3)	0.0489 (11)
C2	−0.0448 (5)	1.1303 (3)	0.3834 (3)	0.0610 (15)
C3	0.1791 (5)	1.3052 (3)	0.1653 (3)	0.0565 (13)
C4	0.1384 (4)	1.3009 (3)	0.0374 (3)	0.0509 (12)
C5	0.2958 (5)	1.1914 (4)	0.0798 (4)	0.0719 (18)
C6	0.0207 (5)	1.0599 (3)	0.1398 (4)	0.0629 (14)
C7	0.0147 (7)	1.1782 (4)	0.0319 (4)	0.085 (2)
C8	0.2064 (7)	1.0878 (5)	0.0496 (5)	0.105 (3)
C9	−0.1206 (4)	1.4364 (3)	0.2402 (3)	0.0548 (13)
C10	−0.2765 (5)	1.4791 (4)	0.3638 (3)	0.0605 (14)
C11	−0.0727 (5)	1.4629 (3)	0.3555 (4)	0.0637 (15)
C12	−0.1630 (5)	1.2362 (4)	0.5233 (3)	0.0634 (16)
C13	−0.3032 (6)	1.3788 (4)	0.5050 (3)	0.0651 (16)
C14	−0.1013 (6)	1.3633 (4)	0.5051 (3)	0.0714 (17)
C15	0.3393 (4)	1.4593 (3)	0.3503 (2)	0.0438 (10)
C16	0.4023 (5)	1.4374 (3)	0.3984 (3)	0.0593 (14)
H16	0.4727	1.4416	0.3820	0.071*
C17	0.3596 (7)	1.4094 (4)	0.4707 (3)	0.083 (2)
H17	0.4012	1.3943	0.5034	0.099*
C18	0.2561 (7)	1.4041 (4)	0.4937 (3)	0.092 (3)
H18	0.2277	1.3849	0.5423	0.111*
C19	0.1934 (6)	1.4261 (4)	0.4475 (4)	0.092 (3)
H19	0.1227	1.4227	0.4644	0.111*
C20	0.2361 (5)	1.4538 (4)	0.3745 (3)	0.0649 (16)
H20	0.1940	1.4685	0.3422	0.078*
C21	0.4861 (3)	1.4046 (2)	0.2128 (2)	0.0348 (8)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C22	0.4585 (4)	1.3764 (3)	0.1632 (3)	0.0463 (11)
H22	0.3964	1.4035	0.1491	0.056*
C23	0.5236 (5)	1.3079 (3)	0.1347 (3)	0.0581 (13)
H23	0.5050	1.2888	0.1015	0.070*
C24	0.6157 (5)	1.2676 (3)	0.1549 (3)	0.0574 (13)
H24	0.6596	1.2219	0.1348	0.069*
C25	0.6430 (4)	1.2943 (3)	0.2046 (3)	0.0559 (13)
H25	0.7045	1.2662	0.2191	0.067*
C26	0.5796 (4)	1.3628 (3)	0.2333 (3)	0.0469 (11)
H26	0.5991	1.3814	0.2664	0.056*
C27	0.4759 (4)	1.5521 (3)	0.2529 (2)	0.0422 (10)
C28	0.4221 (5)	1.6142 (3)	0.2962 (3)	0.0518 (12)
H28	0.3522	1.6187	0.3279	0.062*
C29	0.4735 (6)	1.6683 (4)	0.2913 (4)	0.0698 (17)
H29	0.4375	1.7101	0.3190	0.084*
C30	0.5774 (6)	1.6611 (4)	0.2459 (4)	0.0729 (18)
H30	0.6120	1.6975	0.2435	0.088*
C31	0.6304 (5)	1.6004 (4)	0.2042 (4)	0.0652 (16)
H31	0.7010	1.5956	0.1738	0.078*
C32	0.5798 (4)	1.5460 (3)	0.2068 (3)	0.0485 (11)
H32	0.6158	1.5054	0.1775	0.058*
C33	0.4001 (3)	1.6273 (2)	0.1028 (2)	0.0326 (8)
C34	0.4827 (3)	1.5738 (2)	0.0583 (2)	0.0375 (9)
H34	0.4720	1.5277	0.0549	0.045*
C35	0.5802 (4)	1.5887 (3)	0.0191 (3)	0.0456 (10)
H35	0.6353	1.5528	−0.0105	0.055*
C36	0.5955 (4)	1.6568 (3)	0.0242 (3)	0.0516 (12)
H36	0.6608	1.6672	−0.0029	0.062*
C37	0.5153 (4)	1.7099 (3)	0.0687 (3)	0.0538 (12)
H37	0.5272	1.7553	0.0726	0.065*
C38	0.4170 (4)	1.6954 (3)	0.1077 (3)	0.0435 (10)
H38	0.3623	1.7315	0.1373	0.052*
C39	0.1805 (3)	1.6837 (2)	0.1975 (2)	0.0388 (9)
C40	0.1509 (5)	1.6888 (4)	0.2721 (3)	0.0663 (16)
H40	0.1770	1.6470	0.3036	0.080*
C41	0.0813 (6)	1.7571 (5)	0.2999 (4)	0.093 (2)
H41	0.0605	1.7612	0.3503	0.111*
C42	0.0429 (6)	1.8188 (4)	0.2536 (4)	0.086 (2)
H42	−0.0007	1.8652	0.2725	0.103*
C43	0.0681 (5)	1.8125 (3)	0.1802 (4)	0.0717 (17)
H43	0.0384	1.8534	0.1495	0.086*
C44	0.1378 (4)	1.7453 (3)	0.1520 (3)	0.0542 (13)
H44	0.1565	1.7412	0.1016	0.065*
C45	0.2286 (3)	1.5750 (2)	0.0942 (2)	0.0370 (9)
C46	0.1661 (4)	1.5234 (3)	0.1194 (3)	0.0447 (10)
H46	0.1541	1.5003	0.1678	0.054*
C47	0.1225 (4)	1.5070 (4)	0.0721 (3)	0.0593 (14)
H47	0.0814	1.4723	0.0887	0.071*
C48	0.1392 (5)	1.5418 (4)	0.0001 (3)	0.0633 (15)
H48	0.1089	1.5304	−0.0313	0.076*
C49	0.2000 (5)	1.5928 (3)	−0.0253 (3)	0.0592 (14)
H49	0.2107	1.6163	−0.0736	0.071*
C50	0.2451 (4)	1.6090 (3)	0.0215 (3)	0.0448 (10)
H50	0.2871	1.6431	0.0042	0.054*
C51	0.5513 (3)	1.8687 (3)	0.3999 (2)	0.0374 (9)
C52	0.5214 (4)	1.8030 (3)	0.4065 (3)	0.0512 (12)

Table 2: (continued)

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
H52	0.5673	1.7630	0.3783	0.061*
C53	0.4220 (5)	1.7966 (4)	0.4559 (3)	0.0697 (17)
H53	0.4018	1.7522	0.4607	0.084*
C54	0.3547 (5)	1.8553 (4)	0.4969 (3)	0.0727 (18)
H54	0.2883	1.8509	0.5294	0.087*
C55	0.3836 (4)	1.9207 (4)	0.4910 (3)	0.0666 (16)
H55	0.3367	1.9605	0.5193	0.080*
C56	0.4820 (4)	1.9281 (3)	0.4432 (3)	0.0499 (11)
H56	0.5019	1.9723	0.4399	0.060*
C57	0.7501 (4)	1.8034 (3)	0.2843 (3)	0.0450 (10)
C58	0.7559 (6)	1.8086 (4)	0.2110 (3)	0.0739 (18)
H58	0.7178	1.8541	0.1895	0.089*
C59	0.8156 (8)	1.7491 (6)	0.1690 (5)	0.107 (3)
H59	0.8182	1.7539	0.1196	0.128*
C60	0.8704 (7)	1.6839 (6)	0.1997 (6)	0.107 (3)
H60	0.9095	1.6430	0.1712	0.129*
C61	0.8716 (7)	1.6750 (4)	0.2696 (7)	0.112 (3)
H61	0.9123	1.6292	0.2889	0.135*
C62	0.8096 (6)	1.7366 (3)	0.3146 (4)	0.084 (2)
H62	0.8094	1.7317	0.3636	0.101*
C63	0.7508 (4)	1.8936 (3)	0.3907 (3)	0.0445 (10)
C64	0.8164 (4)	1.9425 (3)	0.3630 (3)	0.0508 (12)
H64	0.8173	1.9723	0.3178	0.061*
C65	0.8804 (5)	1.9461 (5)	0.4034 (5)	0.082 (2)
H65	0.9255	1.9781	0.3850	0.099*
C66	0.8781 (6)	1.9036 (7)	0.4696 (5)	0.116 (4)
H66	0.9207	1.9072	0.4966	0.139*
C67	0.8136 (7)	1.8552 (7)	0.4972 (4)	0.127 (4)
H67	0.8140	1.8255	0.5423	0.152*
C68	0.7479 (5)	1.8503 (5)	0.4586 (3)	0.086 (2)
H68	0.7027	1.8184	0.4778	0.103*
C69	0.4969 (4)	2.1013 (3)	0.2854 (3)	0.0452 (10)
C70	0.5405 (4)	2.1243 (3)	0.3297 (3)	0.0557 (13)
H70	0.6055	2.0933	0.3380	0.067*
C71	0.4888 (5)	2.1924 (3)	0.3614 (4)	0.0661 (15)
H71	0.5188	2.2074	0.3908	0.079*
C72	0.3917 (5)	2.2387 (3)	0.3493 (4)	0.0711 (18)
H72	0.3565	2.2849	0.3706	0.085*
C73	0.3478 (5)	2.2167 (4)	0.3065 (4)	0.0749 (19)
H73	0.2824	2.2479	0.2990	0.090*
C74	0.3991 (4)	2.1486 (4)	0.2739 (3)	0.0608 (14)
H74	0.3685	2.1344	0.2445	0.073*
C75	0.6513 (5)	2.0453 (3)	0.1487 (3)	0.0526 (13)
C76	0.6010 (8)	2.0956 (4)	0.1005 (3)	0.091 (3)
H76	0.5257	2.1140	0.1143	0.109*
C77	0.6603 (11)	2.1193 (5)	0.0323 (4)	0.123 (4)
H77	0.6249	2.1524	−0.0006	0.147*
C78	0.7686 (11)	2.0955 (6)	0.0119 (4)	0.117 (4)
H78	0.8079	2.1121	−0.0348	0.140*
C79	0.8216 (8)	2.0472 (7)	0.0593 (4)	0.120 (4)
H79	0.8970	2.0309	0.0455	0.144*
C80	0.7610 (6)	2.0225 (5)	0.1290 (4)	0.086 (2)
H80	0.7964	1.9900	0.1621	0.104*
C81	0.4769 (5)	1.9742 (3)	0.2295 (4)	0.0703 (19)
C82	0.3897 (6)	1.9655 (4)	0.2887 (5)	0.099 (3)
H82	0.3762	1.9845	0.3331	0.119*

Table 2: (continued)

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C83	0.3208 (8)	1.9272 (6)	0.2814 (8)	0.148 (6)
H83	0.2628	1.9194	0.3216	0.177*
C84	0.3387 (11)	1.9021 (6)	0.2166 (13)	0.196 (9)
H84	0.2921	1.8777	0.2121	0.235*
C85	0.4249 (10)	1.9117 (7)	0.1569 (10)	0.186 (8)
H85	0.4350	1.8944	0.1123	0.223*
C86	0.4966 (7)	1.9466 (5)	0.1618 (7)	0.118 (4)
H86	0.5562	1.9518	0.1218	0.141*

Source of material

$\text{Fe}_3(\text{CO})_{12}$ (0.050 g, 0.10 mmol), Na_2Te_2 (0.045 g, 0.15 mmol), and PPNCl (0.345 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 block crystals were isolated by filtration and washed with MeOH and diethyl ether several times. Crystals of $(\text{PPN})_2[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]$ were obtained in 26% yield, based on the Fe metal used.

Comment

Iron carbonyl teluride clusters have attracted attention a lot especially for their structural novelty and diversity [5–9]. Hitherto known homometallic anionic iron tellurido carbonyl clusters are as follows: $[\text{Fe}_2\text{Te}_3(\text{CO})_6]^{2-}$ [6, 7], $[\text{Fe}_4\text{Te}_2(\text{CO})_{14}]^{2-}$ [8], $[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]^{2-}$ [9], $[\text{Fe}_6\text{Te}_{14}(\text{CO})_{12}]^{2-}$ [7], and $[\text{Fe}_8\text{Te}_{10}(\text{CO})_{20}]^{2-}$ [9]. The cation adopted to stabilize the homometallic anionic iron tellurido carbonyl clusters has been limited to Ph_4P^+ only except $(\text{K-crypt})^+$ for $[\text{Fe}_2\text{Te}_3(\text{CO})_6]^{2-}$. Here we report another homometallic iron tellurido carbonyl cluster compound stabilized with bis(triphenylphosphine)iminium, PPN^+ cations. By employing a methanothermal method, $(\text{PPN})_2[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]$ has been synthesized from the reaction between $\text{Fe}_3(\text{CO})_{12}$ and Na_2Te_2 in the presence of PPNCl . It is noteworthy that $(\text{Ph}_4\text{P})_2[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]$ had been synthesized by the solution method, from the reaction between a fivefold excess of $[\text{Fe}(\text{CO})_5]$ and $(\text{Ph}_4\text{P})_2\text{Te}_4$ in DMF at 85 °C [9].

The $[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]^{2-}$ anion in $(\text{PPN})_2[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]$ was revealed to be of the same molecular structure as the one in the $(\text{Ph}_4\text{P})_2[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]$. The molecular structure

of the $[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]^{2-}$ anion could be divided into three fragments, that is, a $\text{Fe}(\text{CO})_2$ central fragment and two $\text{Fe}_2\text{Te}_2(\text{CO})_6$ side fragments. The $\text{Fe}_2\text{Te}_2(\text{CO})_6$ side fragment shows a so-called ‘butterfly’ structure which is often encountered in other iron carbonyl chalcogenide compounds [10, 11]. There exists a metal-metal bond in each $\text{Fe}_2\text{Te}_2(\text{CO})_6$ side fragment, as the $\text{Fe}(2)\text{--Fe}(3)$ and $\text{Fe}(4)\text{--Fe}(5)$ bond distances are 2.602(1) and 2.606(1) Å, respectively, which are quite similar to the $\text{Fe}\text{--Fe}$ bond distance of 2.592(4) Å in the $(\text{Ph}_4\text{P})_2[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]$. The $\text{Te}(1)\text{--Te}(2)$ and $\text{Te}(3)\text{--Te}(4)$ distances are 3.1342(4) and 3.1454(4) Å, respectively, which are also quite similar to the $\text{Te}\text{--Te}$ distance of 3.152(2) Å in the $(\text{Ph}_4\text{P})_2[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]$. As the $\text{Te}\text{--Te}$ distances of these lengths are too long to be assigned as those of ditelluride, Te_2^{2-} ions, which are normally in the range of 2.7–2.8 Å, each butterfly iron atom is supposed to possess the oxidation state of +1. Considering the oxidation state of +2 in the central Fe atom, the charge valence of the $[\text{Fe}_5\text{Te}_4(\text{CO})_{14}]^{2-}$ anion could be described as $[(\text{Fe}^{+2})(\text{Fe}^{+1})_4(\text{Te}^{-2})_4(\text{CO})_{14}]^{2-}$.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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