

Insertion of methylene group into Fe–Te bond: synthesis and characterization of $\{(\text{CO})_3\text{Fe}(\mu\text{-CH}_2)\text{Te}\}_2$

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Abstract

A novel methylene insertion product is reported. The new compound $\{(\text{CO})_3\text{Fe}(\mu\text{-CH}_2)\text{Te}\}_2$ has been isolated from the reaction of the mixed-chalcogenide compound $\text{Fe}_2(\text{CO})_6(\mu\text{-STe})$ with diazomethane. It has been characterized by IR, ^1H , ^{13}C and ^{125}Te NMR spectroscopy. Its structure has been determined by single-crystal X-ray diffraction methods: monoclinic; space group, $P2_1/c$; $a = 6.743(2)$, $b = 10.996(2)$, $c = 18.007(2)$ Å, $\beta = 96.60(2)^\circ$; $V = 1326.3(2)$ Å³; $Z = 4$; $F(000) = 1024$; Mo $K\alpha$ radiation (graphite monochromated); $\mu = 6.53$ mm⁻¹; $T = 293$ K; $D_c = 2.86$ g cm⁻³; $R = 0.065$ ($R_w = 0.064$).

Keywords: Iron; Tellurium

1. Introduction

Transition metal complexes containing single atom ligands derived from certain main groups of the periodic table have evinced considerable interest in recent times [1–3]. The use of main group elements as ligands firstly serve to stabilize polynuclear metal carbonyl frameworks and secondly can act as initial sites of addition in cluster growth reactions. From Group 16 of the periodic table, S has been extensively used for stabilization of unusual structures and as sites on which coordinatively unsaturated metal carbonyl groups can add [4,5]. Interest in the use of Te for similar purpose arose because of the belief that the larger size and greater electropositive character of Te relative to S or Se would yield clusters of different structural and reactivity features than those containing the lighter chalcogen atoms [6–10]. Investigations of the compounds $\text{Fe}_2(\text{CO})_6(\mu\text{-E}_2)$ and $\text{Fe}_3(\text{CO})_9(\mu_3\text{-E})_2$, where E = S, Se or Te, indicate that indeed the Te-bridged compounds frequently display unique reactivity features and the Te-bridged clusters

have a tendency to adopt more open structures [11]. The compounds $\text{Fe}_2(\text{CO})_6(\mu\text{-E}_2)$ are useful starting materials for the addition of organic and inorganic species [12–16]. The additions can be carried out by two general methodologies. In the presence of a suitable base, the dianion $[\text{Fe}_2(\text{CO})_6(\mu\text{-E})_2]^{2-}$ is formed which readily adds organic or inorganic electrophiles. Because of the highly reactive nature of the E–E bond in $\text{Fe}_2(\text{CO})_6(\mu\text{-E}_2)$, reactive organic species, such as the methylene group [17] and certain acetylenes [18–20], and coordinatively unsaturated metal carbonyl species readily add across the E–E bond [21,22]. The products obtained from both types of reaction have the Fe–Fe and Fe–Te bonds intact. Recently, it has been possible to add across the Fe–Fe bond of $\text{Fe}_2(\text{CO})_6(\mu\text{-Se}_2)$ by a method involving initial blocking of the reactive Se sites by the addition of phenylacetylene across the Se–Se bond to form $(\text{CO})_6\text{Fe}_2\{\mu\text{-SeC(Ph)=C(H)}\}$. Thermolysis of the phenylacetylene adduct in the presence of $\text{Cp}_2\text{Mo}_2(\text{CO})_6$ forms the mixed-metal cluster $\text{Cp}_2\text{Mo}_2\text{Fe}_2(\text{CO})_6(\mu_3\text{-Se})_2(\mu_4\text{-Se})$ [23]. Convenient synthesis of the mixed-chalcogenide compounds $\text{Fe}_2(\text{CO})_6(\mu\text{-EE}')$ provides an opportunity to investigate unusual reactivity and structural aspects arising from the presence of two different types of the chalcogen ligand

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in the same molecule [24]. In this paper, we describe the synthesis and characterization of an unusual compound isolated from the reaction of $\text{Fe}_2(\text{CO})_6(\mu\text{-STe})$ with diazomethane.

2. Results and discussion

When $\text{Fe}_2(\text{CO})_6(\mu\text{-STe})$ and freshly prepared diazomethane are stirred at room temperature, the expected methylene-inserted compound $\text{Fe}_2(\text{CO})_6(\mu\text{-SCH}_2\text{Te})$ (**1**) is obtained as the major product, analogous to the formation of $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_2\text{Te})$ from the reaction of $\text{Fe}_2(\text{CO})_6(\mu\text{-Te}_2)$ with diazomethane [17]. However, the mixed-chalcogenide reaction produces several other compounds with minor yields. By accumulating a sufficient amount of one of these minor products from several runs of the reaction, it has been possible to characterize it fully as $\{(\text{CO})_3\text{Fe}(\mu\text{-CH}_2)\text{Te}\}_2$ (**2**). The IR spectrum of **2** in the carbonyl region displays four bands at $\nu = 2072(\text{w})$, $2052(\text{s})$, $2003(\text{s})$ and $1987(\text{m})$ cm^{-1} due to terminal carbonyl groups. NMR spectroscopy indicates non-equivalent CH_2 groups. ^1H NMR spectrum shows AB-type signals for each CH_2 group at $\delta = 4.05$ ($J = 2.5$ Hz), 4.57 ($J = 2.5$ Hz), 5.23 ($J = 4.1$ Hz) and 5.31 ($J = 4.1$ Hz) ppm. The ^{13}C (^1H) NMR spectrum shows two signals at $\delta = 45.4$ (t, $J_{\text{C-H}} = 165.3$ Hz) and 46.8 (t, $J_{\text{C-H}} = 159.3$ Hz) ppm, in addition to six distinct peaks at $\delta = 202.6$, 204.1 , 205.8 , 209.3 , 210.4 and 212.3 ppm due to the six CO groups. The ^{125}Te NMR spectrum of **2** shows a signal at $\delta = -767.3$ ppm (Fig. 1). Close examination of the signal reveals a complex splitting pattern indicative of coupling between Te and four non-equivalent protons.

Dark-red crystals of **2** were obtained from a hexane- CH_2Cl_2 solvent mixture at 0°C and an X-ray diffraction study was undertaken. The PLUTO diagram of the molecular structure of **2** is shown in Fig. 2. The core geometry of **2** consists of a heavy atom square in which the Fe and Te atoms occupy alternate corners. Two Fe-Te bonds are bridged by a CH_2 group, one on each side of the plane defined by the heavy atoms. Each Fe atom has three terminally bonded carbonyl groups. The bonding of each Fe atom to two Te atoms, one CH_2 group and three CO groups gives a distorted octahedral geometry around it.

The Fe-Te bonds in **2** which are bridged by CH_2 groups, (Fe(1)-Te(2), 2.474 $7(24)$ Å; Fe(2)-Te(1), 2.468 $8(23)$ Å) are slightly shorter than the unbridged Fe-Te bonds (Fe(1)-Te(1), 2.485 $0(24)$ Å; Fe(2)-Te(2), 2.487 $9(24)$ Å). Both types of Fe-Te bond of **2** are somewhat shorter than the Fe-Te bond distances of $2.548(2)$ Å and $2.551(3)$ Å in $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_3)_2$ [25], $2.551(2)$ Å and $2.546(2)$ Å in $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_2\text{Te})$ [17], 2.53 Å (average) in $\text{Fe}_3(\text{CO})_9(\mu\text{-Te})_2$ [26], 2.553 Å (average) in $\{[(\text{MeTe})\text{Fe}_2(\text{CO})_6]_2(\mu\text{-TeCH}_2\text{Te})\}$ [27] and 2.523 Å (average) in $\text{Fe}_2(\text{CO})_6\{\mu\text{-TeC}(\text{Ph})=\text{C}(\text{H})\text{Te}\}$ [28]. The Te-Te distance of $3.320(2)$ Å is outside the normal bonding range. The average Fe-Te-Fe angle in **2** (95.9°) is larger than the Fe-Te-Fe angles in $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_2\text{Te})$ (61.1°), $\text{Fe}_2(\text{CO})_6\{\mu\text{-TeC}(\text{Ph})=\text{C}(\text{H})\text{Te}\}$ (61.3°) [28], $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_3)_2$ (62.3°) [25] and $\text{Fe}_2(\text{CO})_6(\mu\text{-Te}(\text{CH}_2)_2\text{Te})$ (62.6°) [29]. The average Te-Fe-Te angle (84.1°) is also larger than the average Te-Fe-Te angles in $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_2\text{Te})$ (75°) [17] and even $\{\text{Fe}_2(\text{CO})_6\}(\mu_4\text{-Te})(\mu_3\text{-Te})\{\text{Ru}_3(\text{CO})_{11}\}$ (79°) [21] in which a large trirutheniumcarbonyl group is inserted into the Te-Te bond of $\text{Fe}_2(\text{CO})_6(\mu\text{-Te}_2)$. The average Te- CH_2 -Fe bridging angle (76.6°) in **2** is smaller than the average Te- CH_2 -Te angles in $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_2\text{Te})$ (92.1°) and S- CH_2 -S bridging angle in $\text{Fe}_2(\text{CO})_6(\mu\text{-SCH}_2\text{S})$ (94.6°).

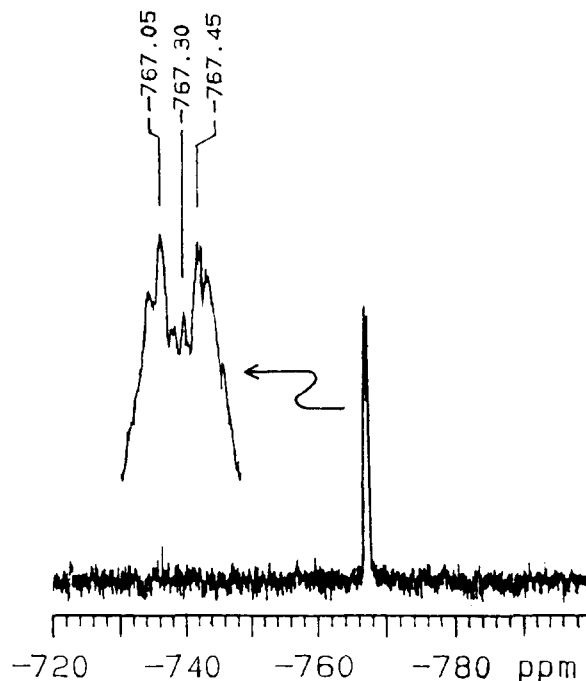


Fig. 1. ^{125}Te NMR spectrum of **2**.

Te CH_2Te] [27] and 2.523 Å (average) in $\text{Fe}_2(\text{CO})_6\{\mu\text{-TeC}(\text{Ph})=\text{C}(\text{H})\text{Te}\}$ [28]. The Te-Te distance of $3.320(2)$ Å is outside the normal bonding range. The average Fe-Te-Fe angle in **2** (95.9°) is larger than the Fe-Te-Fe angles in $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_2\text{Te})$ (61.1°), $\text{Fe}_2(\text{CO})_6\{\mu\text{-TeC}(\text{Ph})=\text{C}(\text{H})\text{Te}\}$ (61.3°) [28], $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_3)_2$ (62.3°) [25] and $\text{Fe}_2(\text{CO})_6(\mu\text{-Te}(\text{CH}_2)_2\text{Te})$ (62.6°) [29]. The average Te-Fe-Te angle (84.1°) is also larger than the average Te-Fe-Te angles in $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_2\text{Te})$ (75°) [17] and even $\{\text{Fe}_2(\text{CO})_6\}(\mu_4\text{-Te})(\mu_3\text{-Te})\{\text{Ru}_3(\text{CO})_{11}\}$ (79°) [21] in which a large trirutheniumcarbonyl group is inserted into the Te-Te bond of $\text{Fe}_2(\text{CO})_6(\mu\text{-Te}_2)$. The average Te- CH_2 -Fe bridging angle (76.6°) in **2** is smaller than the average Te- CH_2 -Te angles in $\text{Fe}_2(\text{CO})_6(\mu\text{-TeCH}_2\text{Te})$ (92.1°) and S- CH_2 -S bridging angle in $\text{Fe}_2(\text{CO})_6(\mu\text{-SCH}_2\text{S})$ (94.6°).

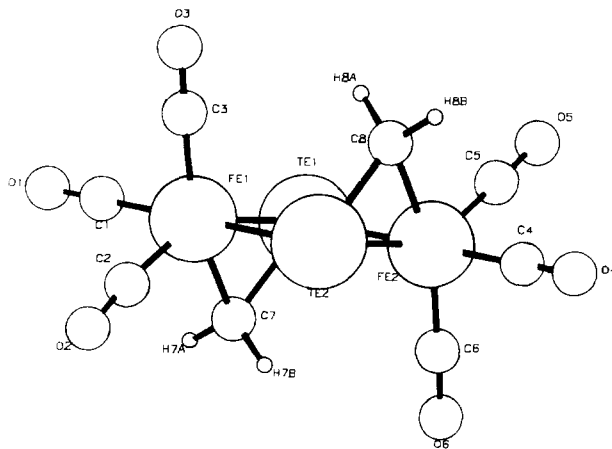
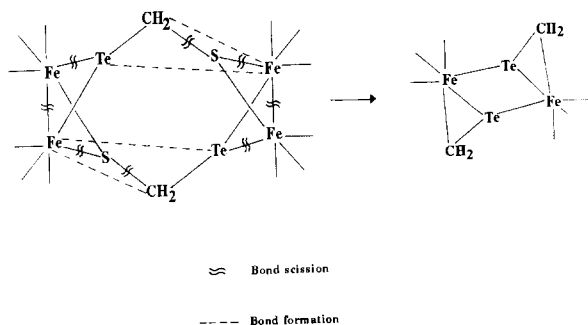


Fig. 2. Molecular structure of **2**.



Scheme 1. Possible intermediate and sequence of bond cleavages and new bond formations during formation of **2**.

The formation of **2** is not observed in the reaction of $\text{Fe}_2\text{Te}_2(\text{CO})_6$ with diazomethane. Scheme 1 depicts a possible sequence of events leading to the formation of **2**. Formation of an intermediate consisting of two Fe_2STe butterfly units linked to each other by two CH_2 groups may be followed by pairwise bond cleavages of Fe–Fe, Fe–Te, Fe–S and S–(CH_2) bonds, accompanied by a pairwise formation of new Fe–Fe and Fe–(CH_2) bonds. Although the intermediate has not been identified in the reaction, attempts are currently under way to isolate and characterize the other minor products formed in the reaction of $\text{Fe}_2(\text{CO})_6(\mu\text{-STe})$ with diazomethane.

3. Experimental details

All the reactions were carried out under pure and dry argon or a nitrogen atmosphere with the use of standard Schlenk techniques. The solvents were purified, dried and distilled under a nitrogen or argon atmosphere prior to use. IR spectra were recorded on a Nicolet 5DXB Fourier transform spectrometer in sodium chloride cells of 0.1 mm path length as hexane solutions. ^1H , ^{13}C and ^{125}Te NMR spectra were obtained on a Varian XL-300 NMR spectrometer in CDCl_3 solutions using appropriate references. The ^{125}Te NMR spectra were referenced to an external standard of Me_2Te ($\delta = 0$ ppm) and the spectra were obtained at the operating frequency of 94.6 MHz; 90° pulses were used with 1.0 s delay and 1.0 s acquisition time. $\text{Fe}_2(\text{CO})_6(\mu\text{-STe})$ was prepared as previously reported [30]. Diazomethane was prepared by dissolving 2.14 g of *N*-methyl *N*-nitrosotoluene *p*-sulphonamide in 30 ml of diethyl ether and cooled in ice. To this a solution of 0.4 g of KOH in 10 ml of 96% ethanol was added. After 5 min, the ethereal solution was distilled from a water bath. The ethereal solution contained 0.32–0.35 g of diazomethane.

3.1. Reaction of $\text{Fe}_2(\text{CO})_6(\mu\text{-STe})$ with diazomethane

To a solution of $\text{Fe}_2(\text{CO})_6(\mu\text{-STe})$ (0.82 mmol) in dry diethyl ether (30 ml) was added dropwise an ethereal solution of diazomethane (24 mmol) (thirtyfold excess) by distilling a basic solution of *N*-methyl *N*-nitroso *p*-toluene sulphonamide in diethyl ether. Addition of diazomethane was continued until thin layer chromatography (TLC) and IR indicated the total consumption of starting material $\text{Fe}_2(\text{CO})_6(\mu\text{-STe})$. The reaction mixture was stirred at room temperature for a further 30 min. The solution was filtered through Celite and the solvent was evaporated. The mixture was redissolved in petroleum ether and subjected to chromatographic work-up on silica gel TLC plates using petroleum ether as the eluent. The light-orange-coloured compound **2** (15 mg, 4.1% based on $\text{Fe}_2(\text{CO})_6(\mu\text{-STe})$) was obtained as the first band, (melting point, 107–109°C (decomposition)). Anal. Found: C, 17.13; H, 0.78 $\text{C}_8\text{H}_4\text{Fe}_2\text{O}_6\text{Te}_2$ calc.: C, 17.05; H, 0.71%.

3.2. X-ray diffraction study of **2**

Single-crystal X-ray data were collected on an Enraf–Nonius CAD-4 diffractometer. The structure was solved by the heavy-atom method, by locating the heaviest Te atoms and subsequent difference Fourier maps revealed the entire structure. Structure was refined by full-matrix least-squares methods using NRCVAX programs [31] to $R = 0.065$ and $R_w = 0.064$ for 1613 ($|F_o| \geq 3\sigma |F_o|$) reflections from a set of 2082 unique data collected on CAD-4 diffractometer. Final ΔF synthesis showed no features above 1.21 electrons \AA^{-3} . Crystal data are given in Table 1. Atomic coordinates, bond lengths and bond angles are given in Tables 2 and 3. General displacement parameters, bond distances and

Table 1
Crystal data

Molecular formula	$\text{C}_8\text{H}_4\text{O}_6\text{Fe}_2\text{Te}_2$
Molecular weight	570.98
Colour; habit	Dark red; square
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit-cell dimensions	
<i>a</i> (Å)	6.743(2)
<i>b</i> (Å)	10.996(2)
<i>c</i> (Å)	18.007(2)
Volume (Å ³)	1326.3(2)
<i>Z</i>	4
Calculated density D_c (g cm ⁻³)	2.86
Absorption coefficient μ (mm ⁻¹)	6.53
<i>F</i> (000)	1024

Table 2

Atomic parameters x , y , z and B_{iso} where the estimated standard deviations refer to the last digit printed

Atom	x	y	z	B_{iso} (Å)
Te(1)	0.0058(2)	0.4484(1)	0.22821(8)	4.24(6)
Te(2)	0.4894(2)	0.4026(1)	0.27824(7)	4.11(6)
Fe(1)	0.2007(3)	0.4088(2)	0.3515(1)	1.43(8)
Fe(2)	0.2933(3)	0.4416(2)	0.1549(1)	1.36(8)
O(1)	-0.137(2)	0.422(1)	0.4367(6)	4.0(6)
O(2)	0.390(3)	0.217(1)	0.4464(7)	5.5(8)
O(3)	0.376(2)	0.631(1)	0.4168(8)	5.4(7)
O(4)	0.636(2)	0.430(1)	0.0716(6)	3.6(6)
O(5)	0.089(2)	0.622(1)	0.0550(7)	4.9(7)
O(6)	0.124(2)	0.213(1)	0.0914(8)	4.7(7)
C(1)	-0.017(3)	0.419(1)	0.4046(7)	2.5(7)
C(2)	0.320(3)	0.293(2)	0.4123(9)	3.6(8)
C(3)	0.310(3)	0.546(2)	0.3922(8)	2.9(7)
C(4)	0.512(3)	0.433(1)	0.1018(7)	2.3(7)
C(5)	0.166(3)	0.553(1)	0.0934(8)	2.8(7)
C(6)	0.191(3)	0.301(1)	0.1156(10)	2.8(7)
C(7)	0.051(2)	0.290(1)	0.2789(7)	4.3(6)
C(8)	0.436(2)	0.562(1)	0.2273(8)	5.1(7)

Table 3

Bond lengths (Å) and bond angles (°) with estimated standard deviations in parentheses

Bond lengths			
Te(1)–Fe(1)	2.4850(24)	Fe(2)–C(4)	1.850(16)
Te(1)–Fe(2)	2.4688(23)	Fe(2)–C(5)	1.804(16)
Te(1)–C(7)	1.979(14)	Fe(2)–C(6)	1.808(17)
Te(2)–Fe(1)	2.4747(24)	Fe(2)–C(8)	2.019(14)
Te(2)–Fe(2)	2.4879(24)	O(1)–C(1)	1.049(19)
Te(2)–C(8)	1.989(16)	O(2)–C(2)	1.114(22)
Fe(1)–C(1)	1.843(16)	O(3)–C(3)	1.102(20)
Fe(1)–C(2)	1.803(18)	O(4)–C(4)	1.047(19)
Fe(1)–C(3)	1.799(15)	O(5)–C(5)	1.110(20)
Fe(1)–C(7)	2.035(13)	O(6)–C(6)	1.132(21)
Bond angles			
Fe(1)–Te(1)–Fe(2)	96.03(8)	Te(1)–Fe(2)–C(4)	178.4(5)
Fe(1)–Te(1)–C(7)	52.8(4)	Te(1)–Fe(2)–C(5)	87.7(6)
Fe(2)–Te(1)–C(7)	97.4(4)	Te(1)–Fe(2)–C(6)	86.9(5)
Fe(1)–Te(2)–Fe(2)	95.80(8)	Te(1)–Fe(2)–C(8)	88.8(4)
Fe(1)–Te(2)–C(8)	96.3(5)	Te(2)–Fe(2)–C(4)	94.5(5)
Fe(2)–Te(2)–C(8)	52.2(4)	Te(2)–Fe(2)–C(5)	146.3(5)
Te(1)–Fe(1)–Te(2)	84.05(7)	Te(2)–Fe(2)–C(6)	110.0(6)
Te(1)–Fe(1)–C(1)	94.7(5)	Te(2)–Fe(2)–C(8)	51.1(4)
Te(1)–Fe(1)–C(2)	145.0(6)	C(4)–Fe(2)–C(5)	93.9(7)
Te(1)–Fe(1)–C(3)	111.5(6)	C(4)–Fe(2)–C(6)	92.7(7)
Te(1)–Fe(1)–C(7)	50.7(4)	C(4)–Fe(2)–C(8)	91.0(6)
Te(2)–Fe(1)–C(1)	177.8(5)	C(5)–Fe(2)–C(6)	102.1(8)
Te(2)–Fe(1)–C(2)	88.9(6)	C(5)–Fe(2)–C(8)	96.3(7)
Te(2)–Fe(1)–C(3)	86.1(5)	C(6)–Fe(2)–C(8)	161.0(7)
Te(2)–Fe(1)–C(7)	90.0(4)	Fe(1)–C(1)–O(1)	177.2(16)
C(1)–Fe(1)–C(2)	93.2(8)	Fe(1)–C(2)–O(2)	175.6(18)
C(1)–Fe(1)–C(3)	92.7(7)	Fe(1)–C(3)–O(3)	179.6(15)
C(1)–Fe(1)–C(7)	90.6(6)	Fe(2)–C(4)–O(4)	179.1(15)
C(2)–Fe(1)–C(3)	102.2(9)	Fe(2)–C(5)–O(5)	179.3(16)
C(2)–Fe(1)–C(7)	95.1(7)	Fe(2)–C(6)–O(6)	179.1(15)
C(3)–Fe(1)–C(7)	162.2(7)	Te(1)–C(7)–Fe(1)	76.5(5)
Te(1)–Fe(2)–Te(2)	84.12(7)	Te(2)–C(8)–Fe(2)	76.7(5)

angles and the structure factor tables are available from the authors.

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