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Reactions of Coordinated Molecules. XXVII. The Crystal and Molecular Structure of [*fac*-$(OC)_3Re(CH_3CO)_3$]BCl: A Triacetylmetalate Complex of Boron

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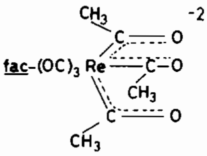
The (triacetyltricarbonylrhenato)boron chloride complex, [fac-$(OC)_3Re(CH_3CO)_3$]*BCl, is characterized structurally by X-ray crystallography. The space group is* $P2_1/m$ *with* Z = 2, *and the unit cell dimensions are:* a = *7.144(2)* Å, b = *8.993(2)* Å, c = *11.134(4)* Å, β = *99.24(2)°* *and* V = *706* Å^3. *The molecule has crystallographically imposed mirror symmetry. Significant crystal decomposition occurred during data collection and resulted in a poorly resolved structure. The final* R *factor is 9.9%, and the weighted* R_w *factor is 8.4%. Typical standard deviations of bond distances and bond angles are* ca. *0.04* Å *and 3°, respectively. The molecule possesses idealized* C_{3v} *symmetry at the* $\pm 3\sigma$ *limit, but only* C_s *symmetry at a* $\pm 2\sigma$ *limit. The possibility of excessive thermal vibrations or disorder is discussed. The structure does confirm that the triacetylrhenate ligand acts as a trioxygen, vicinal, bifurcated chelating ligand when it is coordinated to a boron atom.*

Introduction

In an earlier communication we reported the preparation of the triacetyltricarbonylrhenate dianion [*fac*-$(OC)_3Re(CH_3CO)_3]^{-2}$, *1*, and its coordination to Al(III) and Hf(IV) metal ions [2]. This dianion coordinates to metal ions presumably as a symmetrical, tridentate chelating ligand as shown in *2*. Subsequently, we reported a general preparative route for the synthesis of these metalla ligands and their coordination complexes, and confirmed their solution-phase structure by demonstrating the existence of geometrical isomerism when one of the alkyl substituents of each ligand is not a methyl group [3].

Recently, we reported that the dianion *1* reacts with the boron trihalides to afford the neutral (*fac*-triacetyltricarbonylrhenate)BX complexes, *3*, where X is F, Cl, Br or I [4]. When the boron-bromide compound is treated with excess $AgBF_4$ in primary alcohol solutions the corresponding alkoxy complexes are formed. Presumably, the complexes of type *3* are the only known boron complexes having trioxygen, vicinal, bifurcated chelating ligands [5]. The tripodal rhena ligand formally contains two anionic oxygen donor atoms and one neutral oxygen donor atom; however, electronic delocalization within the rhena moiety may alter the electronic environment of the boron atom quite considerably from the classical neutral, four-coordinate boron-adduct structure to afford a symmetrical structure.



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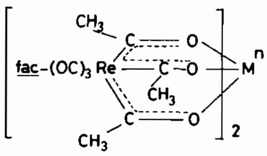

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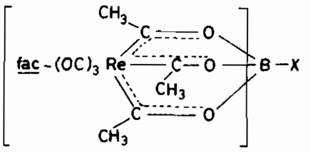

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We wish to report the structural determination of complex *3*, where X is chlorine, by X-ray crystallography to verify the mode of coordination of the triacetyltricarbonylrhenate ligand and to determine the extent of electronic delocalization within the metalla ligand. This is the first crystallographic study of a complex containing a triacylmetalate ligand.

Experimental

Yellow crystals of *3* (X = Cl) were crystallized from a pentane solution at –20 °C by D. T. Hobbs.

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The collection of the X-ray data, the Lorentz, polarization and absorption corrections including a correction for intensity changes of the standard reflections were performed by Molecular Structure Corporation, College Station, Texas, as a commercial, technical service.

A pale yellow crystal of dimensions 0.2 × 0.2 × 0.3 mm was mounted in a capillary. Preliminary examination on the diffractometer showed monoclinic symmetry. The systematic absences indicated the space groups $P2_1$ or $P2_1/m$. Cell constants at 23 ± 1 °C obtained from the computer-centering of 25 reflections were: a = 7.144(2) Å, b = 8.993(2) Å, c = 11.134(4) Å, β = 99.24(2)°. For V = 706 Å^3, mol. wt. = 445.63, and assuming 2 molecules per unit cell, the calculated density is 2.10 g·cm^{-3}.

The data were collected using an Enraf Nonius CAD4 diffractometer and Mo Kα (λ = 0.170730 Å) radiation which was filtered by a graphite crystal incident beam monochromator. Intensity measurements were made using omega-theta scans with an omega-to-theta scan speed ratio of 3.0. Unique data with 2θ up to 45° were collected with a scan range having omega width of 2.0° + 0.35 [tan (theta)] and theta width of 0.33° (omega width). Three standard reflections were measured periodically and 25% decomposition was observed over the data collection. A correction for changes in intensity of the standard reflections was applied to the data. Intensities and standard deviations on intensities were calculated using the formulae: $I = S(C - RB)$ and $\sigma(I) = [S^2(C + R^2B) + (pI)^2]^{1/2}$, where S is the scan rate, C is the total integrated peak count, R is the ratio of scan time to background counting time, B is the total background count and p is a factor introduced to downweight intense reflections and was set to 0.05. Lorentz and polarization corrections were applied to the data. An extinction correction was not made, and an absorption correction based on a series of psi scans was applied to the data. Relative transmission coefficients ranged from 0.68 to 1.00 with an average value of 0.88. The number of independent reflections collected was 988.

A sharpened Patterson function indicated the Re position which provided phases for a difference synthesis that revealed the remaining atoms. This map revealed the location of the Re, B and Cl atoms as well as an acetyl and a carbonyl ligand on a mirror plane at y = ¼. False symmetry was not evident, and the successful location and refinement of the atoms distant from this mirror plane precluded false symmetry and identified the correct space group as $P2_1/m$. A ring of electron density around the C(3) and C(4) methyl carbon atoms indicated rotational disorder, and the H atoms of the disordered methyl groups were approximated by 6 equally spaced and appropriately weighted atoms tetrahedrally located 1.0 Å from the carbon position.

The final refinement was carried out with all non-hydrogen atoms anisotropic and with the isotropic hydrogen atoms included but not refined. The quantity minimized was $\Sigma w(|F\sigma| - |Fc|)^2$ where $w = 1/\sigma(F_o)$. Atomic scattering factors for the neutral atoms were those tabulated by Cromer and Mann [6] except for hydrogen where Stewart's values [7] were used. The anomalous dispersion factors for all atoms except hydrogen were those given by Cromer and Liberman [8]. The final R factor was 0.099 and the weighted R factor, $R_w = \{[\Sigma w(|F\sigma| - |Fc|)^2]/\Sigma w|F\sigma|^2\}^{1/2}$, was 0.084, both calculated with all 988 reflections. The maximum shift to error ratio for the atomic parameters in the final cycle was 0.35. The maximum electron density on the final difference map was 1.74 e Å^{-3} near Re. The atomic parameters are displayed in Table I. Except as noted, calculations were carried out with the X-RAY 67 programs [9] as implemented and updated on the Vanderbilt DEC-1099 computer. A table of observed and calculated structure factors is available from the authors upon request.

Results and Discussion

The crystal structure consists of discrete monomeric molecular units of [*fac*-$(OC)_3Re(CH_3CO)_3$]BCl. The atomic numbering scheme and a perspective view of the molecule are provided in Fig. 1. There are no abnormally short intermolecular contacts. The intermolecular O(2)···H(311) distance of 2.79(2) Å is the shortest such contact, and this distance represents a normal van der Waals contact. Interatomic distances and angles are compiled in

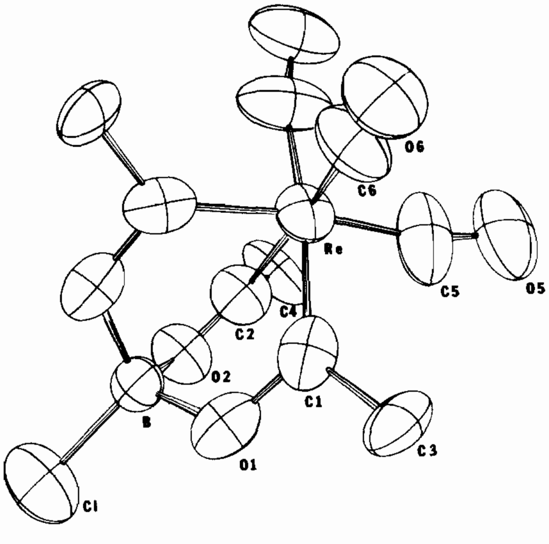


Fig. 1. An ORTEP view of [*fac*-$(OC)_3Re(CH_3CO)_3$]BCl (ellipsoids at 30% probability) showing the atomic numbering scheme.

TABLE I. Atomic Positional and Thermal Parameters of [*fac*-$(OC)_3Re(CH_3CO)_3$]BCl.[a]

Atom	x	y	z	B_{11} or B	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Re	0.42337(22)	¼	0.21671(13)	7.44(9)	9.83(12)	6.13(8)	−1.52	−0.97(6)	−0.29
B	0.3444(48)	¼	0.4829(31)	6(2)	7(2)	6(2)	0	2(2)	0
Cl	0.3015(14)	¼	0.63847(89)	8.3(6)	18(2)	7.7(6)	0	1.0(5)	0
C(1)	0.2868(33)	0.1025(27)	0.2955(27)	8(2)	7(2)	13(2)	3(2)	−7(1)	−3(2)
C(2)	0.6051(47)	¼	0.3855(36)	6(2)	12(3)	9(3)	0	1(2)	0
C(3)	0.1891(37)	−0.0454(24)	0.2596(25)	12(2)	5(2)	12(2)	0(2)	−2(2)	−1(2)
C(4)	0.8169(53)	¼	0.3947(31)	8(3)	25(6)	6(2)	0	2(2)	0
C(5)	0.5491(58)	0.1075(45)	0.1577(33)	18(4)	17(4)	13(3)	11(3)	−6(3)	−6(3)
C(6)	0.2149(57)	¼	0.0758(40)	8(3)	16(4)	10(3)	0	1(2)	0
O(1)	0.2639(20)	0.1161(18)	0.4186(16)	6.6(8)	6.9(9)	10(1)	0.1(7)	−0.6(8)	0.3(9)
O(2)	0.5506(27)	¼	0.4848(20)	5(1)	11(2)	7(2)	0	−1(1)	0
O(5)	0.6541(39)	0.0134(39)	0.1157(21)	17(3)	22(3)	11(2)	6(3)	−4(2)	−4(2)
O(6)	0.0890(41)	¼	−0.0041(27)	12(2)	15(3)	11(2)	0	−6(3)	0
H(311)	0.2653	−0.1318	0.2964	10.8					
H(321)	0.0578	−0.0518	0.2837	10.8					
H(331)	0.1700	−0.0577	0.1671	10.8					
H(312)	0.1587	−0.1032	0.3311	10.8					
H(322)	0.0635	−0.0291	0.2017	10.8					
H(332)	0.2709	−0.1091	0.2144	10.8					
H(411)	0.8651	0.3408	0.3516	14.3					
H(421)	0.8871	¼	0.4801	14.3					
H(412)	0.8798	0.3408	0.4373	14.3					
H(422)	0.8578	¼	0.3088	14.3					

[a] Numbers in parentheses are estimated standard deviations in the least significant digit in this and all other Tables. The anisotropic temperature parameters are of the form $\exp[-\frac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

TABLE II. Interatomic Distances (A) for [*fac*-$(OC)_3Re(CH_3CO)_3$]BCl.

Re–C(1)	1.93(3)	C(2)–C(4)	1.50(5)
Re–C(2)	2.10(4)	C(1)–O(1)	1.42(4)
Re–C(5)	1.75(5)	C(2)–O(2)	1.23(5)
Re–C(6)	1.99(4)	B–O(1)	1.47(3)
C(5)–O(5)	1.27(6)	B–O(2)	1.47(4)
C(6)–O(6)	1.16(5)	B–Cl	1.81(4)
C(1)–C(3)	1.52(4)	O(1)–O(2)	2.39(3)

TABLE III. Interatomic Angles (deg.) for [*fac*-$(OC)_3Re$-$CH_3CO)_3$]BCl.[a]

C(1)–Re–C(2)	83(2)	Re–C(6)–O(6)	178(4)
C(1)–Re–C(5)	90(2)	Re–C(1)–C(3)	137(3)
C(1)–Re–C(6)	89(2)	Re–C(1)–O(1)	122(2)
C(1)–Re–C(1)′	87(2)	Re–C(2)–C(4)	122(3)
C(1)–Re–C(5)′	175(2)	Re–C(2)–O(2)	124(3)
C(2)–Re–C(5)	93(2)	C(4)–C(2)–O(2)	114(3)
C(2)–Re–C(6)	170(2)	C(3)–C(1)–O(1)	102(3)
C(2)–Re–C(1)′	83(2)	C(1)–O(1)–B	116(2)
C(2)–Re–C(5)′	93(2)	C(2)–O(2)–B	117(3)
C(5)–Re–C(6)	94(2)	O(1)–B–O(2)	109(2)
C(5)–Re–C(1)′	175(2)	O(1)–B–O(1)′	110(2)
C(5)–Re–C(5)′	94(2)	O(1)–B–Cl	111(2)
C(6)–Re–C(1)′	89(2)	O(2)–B–O(1)′	109(2)
C(6)–Re–C(5)′	94(2)	O(2)–B–Cl	108(2)
C(1)′–Re–C(5)′	90(2)	O(1)′–B–Cl	111(2)
Re–C(5)–O(5)	174(4)		

[a] The singly primed atoms are generated by the symmetry operation; x, ½ − y, z in this and Table IV.

Tables II and III, respectively, and selected least-squares plane data are listed in Table IV. The values of the interatomic distances and angles were rounded in the normal fashion, while the standard deviations for these values were always rounded to the next larger digit.

When considering the effects of the crystal decomposition and the rather unusual thermal motion of the acetyl-oxygen atoms on the precision of these structural parameters, it is important to not over-interpret the data. However, this structural determination demonstrates clearly that the *fac*-(triacetyltricarbonylrhenate)dianion is coordinated to the boron atom *via* the three acetyl-oxygen atoms, and that this rhenate ligand is acting as a trioxygen, vicinal, bifurcated chelating ligand.

Within a ±3σ limit the complex has idealized C_{3v} symmetry. The molecule has crystallographically-imposed mirror symmetry, and the dihedral angle between the Planes I and II is 119.5° which is very

TABLE IV. Least-Squares Planes , Atomic Deviations and Dihedral Angles for [fac-$(OC)_3Re(CH_3CO)_3$] BCl.

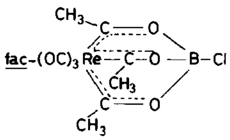
Atom	dev, A	Atom	dev, A
Plane I:	1.676×10^{-7} I + J + 1.966×10^{-8} K = 2.248		
Re	0		
C(2)	0		
O(2)	0		
B	0		
Plane II:	0.8206 I – 0.4928 J + 0.2895 K = 1.743		
Re	0.003	C(2)	1.36
C(1)	–0.007	C(1)′	–1.31
O(1)	0.008	O(2)	1.21
B	–0.004	O(1)′	–1.18
Plane III:	0.6107 I + 1.508×10^{-5} J + 0.7919 K = 3.496		
Re	0	C(5)	0.10
C(1)	0	C(5)′	0.10
C(1)′	0		
Plane IV:	0.9615 I + 8.729×10^{-6} J + 0.2749 K = 2.359		
C(1)	0		
O(1)	0		
C(1)′	0		
O(1)′	0		
Plane V:	–0.8378 I + 6.127×10^{-6} J + 0.5460 K = 1.558		
B	0		
O(1)	0		
O(1)′	0		
Plane VI:	–0.6093 I + 0.7220 J + 0.3279 K = 0.7973		
Re	0	C(6)	0.25
C(1)	0	C(5)′	0.10
C(2)	0		
Plane VII:	–0.4495 I + 0.8665 J – 0.2172 K = –0.5967		
C(1)	0.007	C(3)	–0.77
O(1)	–0.008	C(4)	–0.70
C(2)	–0.008		
O(2)	0.009		
Plane VIII:	-1.331×10^{-2} I – 0.5020 J + 0.8648 K = 3.438		
B	0		
O(1)	0		
O(2)	0		

Dihedral Angles between Planes (deg.)

Planes		Planes	
I–II	119.5	VI–VII	145.9
III–IV	143.6	VII–VIII	128.1
IV–V	131.0		

close to the expected value of 120°. The rhenium atom has an essentially octahedral coordination geometry with the average values for the angles defining the principle coordination axes and for the angles between adjacent ligands being 173(2)° and 90(2)°, respectively. Also, the atoms C(5) and C(5)′ lie only 0.10 Å from Plane III, and C(5)′ is equally close to Plane VI. However, atom C(6) lies 0.25 Å from Plane VI which might reflect the actual lower symmetry of the rhenium coordination geometry.

The elucidation of the intramolecular electronic structure of this complex is very difficult at this level of precision. The two extreme bonding formulations are the delocalized structure, *4*, and the Lewis acid–base, adduct structure, *5*. The solution-phase structure, as revealed by the IR and PMR spectra at room temperature, is consistent with the C_{3v} symmetry of structure *4* [4]. Although a rapid dynamic exchange among the three degenerate isomers of *5* would be revealed as a symmetrical structure on the PMR timescale, it is rather unlikely that such a process would be fast on the IR timescale, as well. However, the expected splitting of the carbonyl ligand C–O stretching vibrational E band in going from *4* to *5* may be too slight to be resolved.



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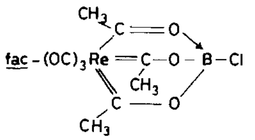


5

At a ±2σ limit, the structural data indicate a localized electronic structure as shown in *5*. The C(1)–C(3) and C(2)–C(4) distances of 1.52(4) Å and 1.50(5) Å, respectively, represent normal $C(sp^2)$–$C(sp^3)$ single bonds. The sum of the angles centered at C(1) and C(2) total to 360(3)° indicating a planar hybridization geometry about these acyl carbon atoms.

The Re–C(2) and C(2)–O(2) distances of 2.10(4) Å and 1.23(5) Å respectively, identifies the acyl group which lies in the crystallographic mirror plane as the unique acetyl ligand in structure *5*. The corresponding Re–C and acetyl C–O distances in the acetylaniline complex, *cis*-$(OC)_4Re(CH_3CO)(NH_2Ph)$, *6*, are 2.211(6) Å and 1.214(7) Å, respectively [10]. The slightly shorter Re–C(2) distance may indicate more double-bond character in this bond than in the Re to acyl carbon bonds of typical rhenium–acetyl complexes. The Re–acyl carbon distances in the rhena-acetylacetone molecule, [*cis*-$(OC)_4Re(CH_3CO)_2$]H, *7*, are 2.15(2) Å and 2.18(2) Å [11].

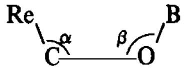
The Re–C(1) and Re–C(1)′ distances of 1.93(3) Å represent rhenium-carbon bonds which have considerable double-bond character. These bonds correspond formally to the rhenium–carbenoid carbon bonds of *5*. The rhenium–carbenoid carbon distance in *cis*-$(OC)_5MnRe(CO)_4[C(OCH_3)(CH_3)]$ is 2.094(7) Å [12], and the usual range of rhenium–terminal carbonyl carbon distances is 1.92–2.00 Å [10, 11]. Based on these literature values the Re–C(1) and Re–C(1)′ bonds in *5* must have a bond order of at least 1.5.

The C(1)–O(1) and C(1)′–O(1)′ distances of 1.42(4) Å are very close to the predicted $C(sp^2)$–O single bond distance of 1.40 Å. A formal bond order of 1.0 for these bonds is consistent with the localized electronic structure of *5*. The average value of the acyl C–O distances in the rhena–acetylacetone molecule [11], *7*, and the bis(rhena–acetylacetonato)-copper complex [13], *8*, are 1.27(2) Å and 1.245(5) Å, respectively. These distances are significantly shorter than the $C(sp^2)$–O single bond distance, and they indicate electronic delocalization within the rhena–acetylacetonate ligands. Presumably, such delocalization within these two acyl groups of the triacetylrhenate ligand of *5* is absent.

The three boron–oxygen distances do not differ significantly. This structural feature probably reflects the bonding tendency of the boron atom to attain a formal sp^3-hybridization geometry. The shorter C(2)–O(2) distance is compensated by the longer Re–C(2) distance, thus permitting the B–O(2) distance to be as long as the B–O(1) and B–O(1)′ distances. Also, there may be a slight tilting of the C(2)–acyl group toward the boron atom since the C(2)–Re–C(1) angle is 4° smaller than the C(1)–Re–C(1)′ angle.

The O(1)–O(2) and O(1)–O(1)′ 'bite' distances are 2.39(3) Å and 2.41(3) Å, respectively. These distances also reflect the bonding preferences of the boron atom. The O–O 'bite' distances in the ferra–β-diketonate and the β-diketonate boron complexes, $[\eta^5\text{-}C_5H_5(OC)Fe(CH_3CO)(i\text{-}C_3H_7CO)]BF_2$, *9*, and (benzoylacetonato)BF_2, *10*, are 2.422(2) Å [14] and 2.456 Å [15], respectively. The average values of the B–O distances in these metalla– and non-metalla–β-diketonate boron difluoride complexes are 1.479(3) Å and 1.487(3) Å, respectively. Similarly, the B–O distances of complex *5* are 1.47(4) Å. Apparently, the three possible pairings of bidentate, chelating oxygen donor atoms within the triacetylrhenate ligand coordinate to the boron atom like the bidentate metalla– and non-metalla–β-diketonate ligands of *9* and *10*. These structural similarities reflect the bonding preferences of the boron atom, since the *intra*-ligand electronic structure varies considerably among these three classes of molecules.

The triacetylrhenate ligand defines three six-membered rings (two of which are symmetry equivalent) formed by the three possible pairings of acetyl groups. One such chelate ring has as backbone atoms; Re–C(1)–O(1)–B–O(2)–C(2). Each ring has a distinct 'boat shaped' structure as shown below:



The angles α and β are determined from the intersection of the Planes III and V with IV and Planes VI and VIII with VII. The methyl substituents lie below the planes defined by the acyl carbon and oxygen atoms. A similar 'boat-shaped' structure is observed for the metalla–β-diketonate ligands of *8* and *9*, and the values of α and β for each complex are shown below:

Complex	α(°)	β(°)
5	143.6	131.0
5	145.9	128.1
8	145.1	142.8
9	156.2	151.8

These data indicate a similarity between the chelate ring structures of the nonplanar metalla–β-diketonate ligand and those of chelate rings of the triacetylrhenate ligand. The more severe bending of the triacetylrhenate ligand at the acyl oxygen atoms, β, probably reflects the tripodal coordination of this ligand to the boron atom.

According to structure *5*, a shortening of the Re–carbonyl carbon bonds which are *trans* to the carbenoid-carbon atoms is expected. This *trans*-effect is observed in that the Re–C(6) distance of 1.99(4) Å is significantly longer than the Re–C(5) and Re–C(5)′ distances of 1.75(5) Å. However, although the Re–C(6)–O(6) geometry is typical of that observed for Re–carbonyl groups, the Re–C(5)–O(5) and Re–C(5)′–O(5)′ geometries have unusually short Re–C distances and unrealistically long C–O distances. It is possible that the positions of C(5) and C(5)′ have been determined poorly.

It is important to note that the initial electron density difference maps did not show any signs of disorder in the positions of the atoms comprising the acetyl ligands. However, Fig. 1 shows that the maximum thermal vibrations for these atoms occur in the planes defined by the acyl C–O and O–B bonds. The thermal motion of the three acetyl oxygen atoms is particularly significant. Since the maximum atomic thermal motion usually occurs along the direction perpendicular to the bonding plane, we propose that the triacetylrhenate ligand is rapidly exchanging among the three isomers having structure *5*. A similar interpretation of this type of thermal motion is well-defined in complexes undergoing a dynamic Jahn–Teller distortion [16]. The presence of such a rapid isomerization in complex *5* may also contribute to the poor resolution of the atomic positions within this molecule.

Acknowledgements

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