Reactions of Oximes with Covalent Halides. Part II.[1] Formation of α-Benzil Monoxime and Benzamide Adducts of Tin Tetra-bromides and -chlorides

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Reaction of α-benzil monoxime, α-bmoH (1 or 2 mol.) and SnX_4 (1 mol.) in benzene followed by immediate removal of the product gave the adducts $SnX_4,n(\alpha\text{-bmoH})$ (X = Cl, n = 1 or 2; X = Br, n = 2). In contrast no reaction was observed between α-bmoH and SnI_4 and between β-bmoH and SnX_4 (X = Cl, Br, or I). The adducts $SnX_4,n(\alpha\text{-bmoH})$ are thermally unstable and when stirred in benzene, or on heating undergo a Beckmann fragmentation to give phenyl cyanide, benzoic anhydride, tin tetrahalide, and the 1:2 adduct of benzamide with tin tetrahalide. The i.r., Raman, and Mössbauer spectra of the 1:2 adducts of benzamide with tin tetrahalide suggest O-bonded benzamide with *trans*-octahedral (X = Cl) or *cis*-octahedral (X = Br) structures.

In Part I[1] of this series it was reported that reactions of the benzil monoximes (α- and β-bmoH) with titanium tetrachloride could lead either to products containing the anionic ligand, bmo$^-$, or to adducts containing the neutral ligands, α- or β-bmoH. We now report the reactions of the benzil monoximes with tin tetrahalides (X = Cl, Br, or I).

TABLE 1

Elemental composition of $SnCl_4,\alpha$-bmoH, $SnBr_4,\alpha$-bmoH, $SnCl_42BzNH_2$, and $SnBr_4,2BzNH_2$

	Calculated %					Found %				
Compound	C	H	X	N	Sn	C	H	X	N	Sn
$SnCl_4,\alpha$-bmoH	34·6	2·3	29·2	2·9	24·4	33·7	2·6	28·5	5·5	23·8
$SnCl_4,2BzNH_2$	33·4	2·8	28·2	5·6	23·6					
$SnBr_4,\alpha$-bmoH	25·3	1·7	48·2	2·1	17·9	24·6	2·1	47·2	4·0	17·2
$SnBr_4,2BzNH_2$	24·7	2·1	47·0	4·1	17·4					

When tin tetrachloride or tetrabromide (1 mol.) and α-benzil monoxime (2 mol.) were mixed in chloroform yellow solutions resulted. After *ca.* 4 hr. a white solid separated from each solution. The elemental analysis, with the exception of nitrogen, of both solids corresponded closely to the 1 : 1 adduct of the respective tetrahalide and α-benzil monoxime (Table 1). Adduct formation was earlier observed when titanium tetrachloride (1 mol.) and α-benzil monoxime (1 or 2 mol.) were mixed in chloroform.[1] Consequently it was tempting to formulate the white solids as SnX_4,α-bmoH. However, the nitrogen content was checked several times and its persistent high value together with the low yield (*ca.* 50% for a 1 : 1 reaction), cast doubts on the validity of this formulation. Hydrolysis of the white solids showed that the organic component was benzamide and the white solids were formulated as

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[1] Part I, J. Charalambous and M. J. Frazer, *J. Chem. Soc.* (*A*), 1968, 2361.

tetrahalogenodibenzamidetin(IV). The analytical data were in good agreement with this formulation (Table 1); further evidence was obtained by comparison of the i.r. spectra with that of authentic samples of tetrahalogenodibenzamidetin(IV) obtained from the reaction of the tetrahalides with benzamide. Subsequent reactions in benzene showed that apart from the benzamide adducts, the system α-benzil monoxime–tin tetrahalide gives also phenyl cyanide and benzoic anhydride. These products arise from the α-benzil monoxime adducts which are formed initially [reactions (1) and (2)] and which could be isolated by removing them from the reaction mixture immediately.

$$\mathrm{SnX_4 + 2\alpha\text{-}bmoH \longrightarrow SnX_4,2\alpha\text{-}bmoH \longrightarrow}$$
$$(\mathrm{X = Cl\ or\ Br})$$
$$\mathrm{0{\cdot}5(SnX_4,2BzNH_2) + PhCN + Bz_2O + 0{\cdot}5SnX_4} \quad (1)$$

$$\mathrm{SnCl_4 + \alpha\text{-}bmoH \longrightarrow SnCl_4,\alpha\text{-}bmoH \longrightarrow}$$
$$\mathrm{0{\cdot}25(SnCl_4,2BzNH_2) + 0{\cdot}5PhCN + 0{\cdot}5Bz_2O + 0{\cdot}75SnCl_4} \quad (2)$$

When tin tetrachloride (1 mol.) and α-benzil monoxime (1 mol.) were mixed in benzene the yellow adduct $\mathrm{SnCl_4,\alpha\text{-}bmoH}$ was formed and isolated by immediate filtration. Similarly tin tetrachloride (1 mol.) and α-benzil monoxime (2 mol.) gave the yellow adduct $\mathrm{SnCl_4,2\alpha\text{-}bmoH}$. Reaction of tin tetrabromide (1 mol.) and α-benzil monoxime (1 or 2 mol.) in benzene gave yellow solutions, from which only the yellow 1 : 2 adduct, $\mathrm{SnBr_4,2\alpha\text{-}bmoH}$, was precipitated by the addition of cyclohexane. The failure to isolate the 1 : 1 adduct is not surprising, since the preferential formation of 1 : 2 adducts has been observed in the reactions of tin tetrabromide with quinolin-8-ol[2] and salicylaldehyde.[3] In contrast to the tetrachloride and tetrabromide, tin tetraiodide and α-benzil monoxime were recovered unchanged after being stirred in benzene. The different behaviour of the tetraiodide is consistent with its weaker acceptor properties.[4]

In the system β-benzil monoxime–$\mathrm{SnX_4}$ (X = Cl, Br, or I) in benzene there was no observable reaction after 6 hr. at room temperature and the starting materials were recovered. This behaviour contrasts with that of titanium tetrachloride which forms both 1 : 1 and 1 : 2 adducts with β-benzil monoxime, but is consistent with the stronger acceptor properties of titanium tetrachloride compared to the tin tetrahalides.[5]

The adducts $\mathrm{SnCl_4,\alpha\text{-}bmoH}$ and $\mathrm{SnX_4,2\alpha\text{-}bmoH}$ were hydrolytically unstable products. The adducts were also thermally unstable and on heating (*ca.* 70°) decomposed to give the benzamide adduct of the tetrahalide, phenyl cyanide, benzoic anhydride, and some tetrahalide. The stoicheiometry of the decomposition [equations (1) and (2)] has been established by isolating quantitatively and characterising all the products.

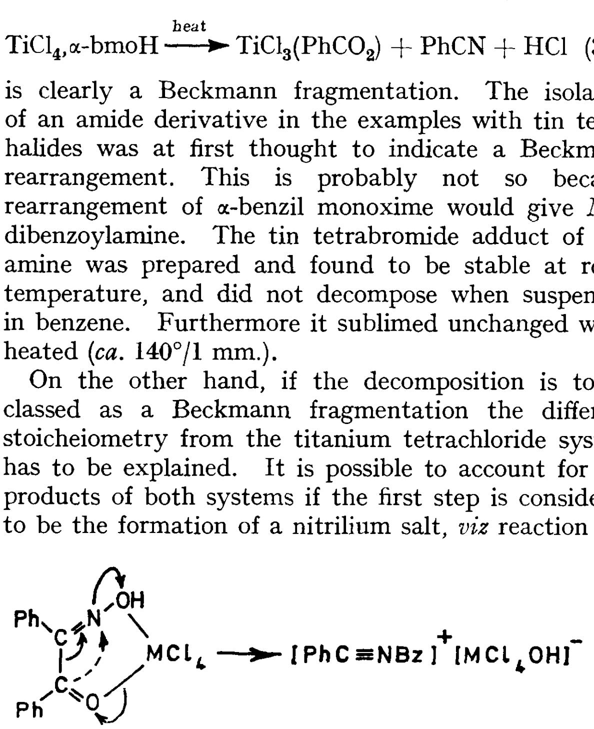
Decomposition of the adducts occurred also on standing at room temperature (2—3 days) and when they were dissolved (X = Br) or suspended (X = Cl) in benzene. The stoicheiometry of decomposition in benzene was again shown to correspond to equations (1) or (2), by isolating and characterising the decomposition products. Furthermore, the decomposition in benzene was monitored by i.r. spectroscopy (1800—1450 cm.$^{-1}$). This clearly illustrated the changes in going from the adduct to the decomposition products, *i.e.* the gradual disappearance of bands characteristic of co-ordinated α-benzil monoxime (1525 cm.$^{-1}$) and the gradual appearance of bands characteristic of benzoic anhydride (1780 and 1735 cm.$^{-1}$) and tetrahalogenobenzamidetin(IV) (1530 cm.$^{-1}$).

The decomposition behaviour of the tin tetrahalide adducts contrasts with that of the titanium tetrachloride–α-benzil monoxime derivatives.[1] The titanium derivatives were stable at room temperature, were unaffected by benzene or chloroform, and decomposed only when heated at higher temperatures (*ca.* 130°). Furthermore, the decomposition of the titanium derivatives led to different products, *viz* reaction (3), and this

$$\mathrm{TiCl_4,\alpha\text{-}bmoH \xrightarrow{heat} TiCl_3(PhCO_2) + PhCN + HCl} \quad (3)$$

is clearly a Beckmann fragmentation. The isolation of an amide derivative in the examples with tin tetrahalides was at first thought to indicate a Beckmann rearrangement. This is probably not so because rearrangement of α-benzil monoxime would give *NN*-dibenzoylamine. The tin tetrabromide adduct of this amine was prepared and found to be stable at room temperature, and did not decompose when suspended in benzene. Furthermore it sublimed unchanged when heated (*ca.* 140°/1 mm.).

On the other hand, if the decomposition is to be classed as a Beckmann fragmentation the different stoicheiometry from the titanium tetrachloride system has to be explained. It is possible to account for the products of both systems if the first step is considered to be the formation of a nitrilium salt, *viz* reaction (4),



(4)

as has been suggested for similar fragmentations with other acids.[6]

When M = Ti the anion would readily lose hydrogen chloride and the resulting species could form $\mathrm{PhCOTiCl_3}$ and PhCN. However when M = Sn hydrogen chloride would not be eliminated from the anion and it is then possible to account for the products of equations (1) and (2) by a somewhat complex series of reaction steps.

The Adducts $\mathrm{SnX_4,2BzNH_2}$ (X = Cl or Br).—Although the adducts $\mathrm{SnX_4,2BzNH_2}$ (X = Cl or Br) have been

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[3] I. Douek and M. J. Frazer, unpublished work.

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prepared earlier[7] very little is known about their structure and properties. Their isolation and synthesis in conjunction with the work discussed earlier has prompted a study of some of their physical properties. These adducts are white solids which are thermally stable but are slowly hydrolysed by cold water. Some spectroscopic (i.r., Raman, and Mössbauer) measurements are in Table 2.

TABLE 2

Spectroscopic data on $SnX_4,2BzNH_2$ (X = Cl or Br) and $BzNH_2$

	X = Cl	X = Br	$BzNH_2$	Assignment
	3435m	3437m	3367m	
	3347m	3345m	3145m	NH
	3265w	3280w		
	1645vs	1640vs	1625s	Amide II
	1600m	1600m	1600s	Aromatic vibrations
	1590w	1590w	1590w	
I.r. (cm.$^{-1}$)	1528vs	1528vs	1658s	Amide I
	375w	375w	255s	
	348sh	343s	210w	
	332vs	325s	195w	
	290m	306s	160sh	
	202w	278sh	150m	
	195s	221m		
	163s	205m		
	153s	168vs		
	130w	155m		
	105m	145w		
	95w	95w		
	314vs	286w	397s	
	290w	210w	244w	
Raman (cm.$^{-1}$)	252m	194vs	181sh	
	197w	155m	176s	
	157w	108w	132vs	
Mössbauer	0·38[a]	0·58[b]		
	0·79[a]	0·40[b]		

[a] δ(mm. sec.$^{-1}$) Relative to tin(IV) oxide. [b] Δ(mm. sec.$^{-1}$)

Insolubility in suitable solvents (benzene, chloroform, nitrobenzene) prevented molecular weight measurements but the observed chemical shifts in their Mössbauer spectra fall in the range where the chemical shifts of octahedral tin(IV) chloride and bromide adducts are frequently found.[8] In the example of tetrachlorodibenzamidetin(IV) the i.r. spectrum shows a strong band at 332 cm.$^{-1}$ and the Raman spectrum an intense line at 314 cm.$^{-1}$ which is non-coincident with the i.r. band. These bands are assignable to Sn–Cl stretching and indicate a *trans*-octahedral geometry for $SnCl_4,2BzNH_2$. The i.r. and Raman spectra of tetrabromodibenzamidetin(IV) are very complex and do not allow unambiguous assignment of Sn–Br vibrations except for a very strong line at 194 cm.$^{-1}$ in the Raman spectrum assignable to Sn–Br stretching. The complexity of the spectra is not compatible with *trans*-geometry and furthermore the i.r. spectrum (400—80 cm.$^{-1}$) is different from the spectrum of *trans*-octahedral $SnBr_4,2py$ (py = pyridine).[9] *cis*-Geometry is, therefore, tentatively proposed for the bromo-complex. Further evidence for the proposed structures of $SnX_4,2BzNH_2$ is provided by Mössbauer spectroscopy. The quadrupole splitting Δ of the chloro-compound is twice that of the bromo-compound; this is in accord with calculations[10] and earlier observations[10,11] which indicate that in octahedral complexes of the type $SnA_4B_2, \Delta_{trans} \sim 2\Delta_{cis}$.

For amide adducts the bonding involves donation from either the oxygen or the nitrogen atom. The i.r. spectra of both adducts show four bands in the region 1550—1700 cm.$^{-1}$ By comparison with the spectrum of the free ligand[12] and the spectra of other amide complexes,[12,13] these bands are assigned as shown in Table 2. The shift of the amide I band to a lower frequency (*ca.* 130 cm.$^{-1}$) and the shift of the amide II band to a higher frequency (*ca.* 20 cm.$^{-1}$) in going from the ligand to the adducts indicates that the bonding of the ligand to tin is probably from the carbonyl oxygen. This is also supported by the shift of the ν(N–H) to higher frequency.[13]

EXPERIMENTAL

Reaction of SnX_4 (X = Cl *or* Br) (1 *mol.*) *with* α-*Benzil Monoxime* (2 *mol.*).—α-Benzil monoxime (3·4 g., 2 mol.) was slowly added to a solution of tin tetrachloride (1·8 g., 1 mol.) in chloroform (*ca.* 150 ml.). A yellow solution resulted from which white tetrachlorodibenzamidetin(IV) (1·7 g., 90%) (Table 1 for analysis, confirmed by i.r. spectrum[7]) separated during 4 hr.; it was filtered off and dried at 20°/1 mm.

Similarly, α-benzil monoxime (4·2 g., 2·0 mol.) and tin tetrabromide (4·0 g., 1·0 mol.) gave white tetrabromodibenzamidetin(IV) (2·6 g., 80%) (Table 1 for analysis, confirmed by i.r. spectrum[7]).

In subsequent experiments concentration of the filtrate, obtained after removal of the tetrahalogenodibenzamidetin(IV) adduct, gave a residue which contained phenyl cyanide and benzoic anhydride as indicated by i.r. spectroscopy.

Preparation of α-*Benzil Monoxime of* SnX_4 (X = Cl *or* Br).—(*a*) $SnCl_4$,α-bmoH. α-Benzil monoxime (2·9 g., 1·0 mol.) was slowly added to a solution of tin tetrachloride (3·6 g., 1·1 mol.) in benzene (*ca.* 150 ml.) to give yellow *tetrachloro*-(α-*benzil monoxime*)*tin*(IV) (4·4 g., 70%) (Found: C, 34·6; H, 2·3; Cl, 29·1; N, 2·9; Sn, 24·1. $C_{14}H_{11}Cl_4NO_2Sn$ requires C, 34·6; H, 2·3; Cl, 29·2; N, 2·9; Sn, 24·4%); this was immediately filtered off, and dried at 20°/1 mm.

(*b*) $SnCl_4$,2α-bmoH. Tin tetrachloride (2·8 g., 1·0 mol.) was slowly added to a suspension of α-benzil monoxime (4·8 g., 2·0 mol.) in benzene (*ca.* 150 ml.) to give yellow *tetrachlorodi*-(α-*benzil monoxime*)*tin*(IV) (5·7 g., 76%) (Found: C, 47·4; H, 3·2; Cl, 19·8; N, 3·9; Sn, 16·6. $C_{28}H_{22}Cl_4N_2O_4Sn$ requires C, 47·3; H, 3·1; Cl, 20·0; N, 3·9; Sn, 16·7%); it was immediately filtered off and dried at 20°/1 mm.

(*c*) $SnBr_4$,2α-bmoH. α-Benzil monoxime (4·8 g., 2·0 mol.) was slowly added to a solution of tin tetrabromide

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(4·7 g., 1·0 mol.) in benzene (*ca.* 150 ml.). A yellow solution resulted to which cyclohexane (*ca.* 100 ml.) was immediately added. Bright yellow *tetrabromodi-*(*α-benzil monoxime*)-*tin*(IV) (9·1 g., 96%) (Found: C, 37·8; H, 2·5; Br, 36·1; N, 3·2; Sn, 13·2. $C_{28}H_{22}Br_4N_2O_4Sn$ requires C, 37·8; H, 2·5; Br, 36·0; N, 3·1; Sn, 13·3%) separated out; it was immediately filtered off and dried at 20°/1 mm.

Tetrabromodi-(α-benzil monoxime)tin(IV) (3·3 g.) was also obtained when tin tetrabromide (4·0 g., 1 mol.) and α-benzil monoxime (2·0 g., 1 mol.) were mixed and treated as above.

Tin Tetraiodide (1·0 *mol.*) *and α-Benzil Monoxime* (n *mol.*) (n = 1 *and* 2).—α-Benzil monoxime (2·0 g., 1·0 mol.) was gradually added to a solution of tin tetraiodide (5·6 g., 1·0 mol.) in benzene (*ca.* 150 ml.) with stirring. After 12 hr., solid α-benzil monoxime (1·8 g., 90%) (mixed m.p. 138°, lit.,[14] m.p. 139°) (confirmed by i.r. spectroscopy) was filtered off and dried at 20°/1 mm. Benzene was removed from the filtrate leaving a residue of tin tetraiodide (5·5 g., 98%) (Found: I, 81·5; Sn, 19·1. Calc. for I_4Sn: I, 81·0; Sn, 18·9%).

Similar recovery of materials was found when tetraiodide (1·0 mol.) and α-benzil monoxime (2·0 mol.) were stirred in benzene for 12 hr.

TABLE 3

Fragmentation of $SnX_4,n(\alpha\text{-bmoH})$

$SnX_4,n(\alpha\text{-bmoH})$ (1·0 mol.)				Products							
				$SnX_4,2BzNH_2$		SnX_4		PhCN		Bz_2O	
X	n	Wt. (g.)	Conditions	(g.)	(mol.)	(g.)	(mol.)	(g.)	(mol.)	(g.)	(mol.)
Cl	1	5·5	*a*	1·0	0·18	1·9	0·65	0·4	0·35	1·1	0·42
Cl	2	6·1	*a*	1·8	0·40	0·9	0·40	0·7	0·80	1·8	0·93
Br	2	5·8	*a*	1·8	0·40	1·1	0·40	0·5	0·73	1·2	0·82
Cl	1	6·0	*b*	1·1	0·18	2·0	0·62	0·5	0·42	1·2	0·42
Cl	2	4·6	*b*	1·5	0·45	0·6	0·35	0·6	0·90	1·3	0·90
Br	2	5·0	*b*	1·6	0·40	0·9	0·35	0·5	0·85	1·1	0·85

[a] Pyrolysis at *ca.* 70°. [b] In benzene at 20°.

Tin Tetrahalide (X = Cl, Br, *and* I) (1·0 *mol.*) *and β-Benzil Monoxime* (n *mol.*) (n = 1 *and* 2).— Solid β-benzil monoxime (4·6 g., 1·0 mol.) was added, with stirring, to a solution of tin tetrachloride (5·3 g., 1·0 mol.) in benzene (*ca.* 100 ml.). After 6 hr. β-benzil monoxime (3·8 g., 83%) (mixed m.p. 114°, lit.,[14] m.p. 114°) (confirmed by i.r. spectroscopy) was filtered off, washed with cyclohexane, and dried at 20°/1 mm. The solvent was removed from the filtrate at 20°/20 mm. and the residue was extracted with cyclohexane. The extract on concentration, gave a residue of tin tetrachloride (4·8 g., 88%) (Found: Cl, 55·0; Sn, 45·3. Calc. for Cl_4Sn: Cl, 54·4; Sn, 45·8%).

Similar recovery of materials was found when β-benzil monoxime (2 mol.) and $SnCl_4$ (1 mol.) or SnX_4 (X = Br or I) (1 or 2 mol.) were stirred in benzene at 20° for 6 hr.

Hydrolysis of Tetrachloro-(*α-benzil monoxime*)*tin*(IV) *and Tetrabromodi-*(*α-benzil monoxime*)*tin*(IV).—Tetrachloro-(α-benzil monoxime)tin(IV) (2·1 g., 1·0 mol.) was dissolved in a mixture of ether and water (*ca.* 100 ml., 5% water); the mixture was stirred for 1 hr. after which the ethereal layer was separated from the resultant mixture; it gave, on concentration, α-benzil monoxime (0·8 g., 0·8 mol.) (Found: C, 74·5; H, 5·0; N, 6·0. Calc. for $C_{14}H_{11}NO_2$: C, 74·7; H, 4·9; N, 6·2%) (mixed m.p. 139°, lit.,[14] m.p. 139°).

Similarly, tetrabromodi-(α-benzil monoxime)tin(IV) (1·7 g., 1·0 mol.) gave α-benzil monoxime (0·7 g., 1·6 mol.) (mixed m.p. 140° lit.,[14] m.p. 139°) (confirmed by i.r. spectroscopy).

Fragmentation of the Adducts $SnX_4,n\alpha$-bmoH (X = Cl, *n* = 1 *or* 2; X = Br, *n* = 2).—(a) *Pyrolysis.* The solid adduct (1 mol.) was heated at 70° for 1 hr. Benzene (*ca.* 100 ml.) was then added and the resultant mixture was stirred for 1 hr. White tetrahalogenodibenzamidetin(IV) (identified by elemental analysis, confirmed by i.r. spectroscopy) was filtered off and dried at 20°/1 mm. Benzene was removed from the filtrate at 20°/20 mm., leaving a brown viscous residue. Tin tetrahalide (identified by analysis) was removed from the residue at 40°/1 mm. and collected at −80° (X = Cl) or as a sublimate (X = Br). Phenyl cyanide (identified by i.r. spectroscopy and g.l.c.) was subsequently removed at 70°/1 mm. and collected at −80°, leaving a residue which contained benzoic anhydride (identified by i.r. spectroscopy) (estimated by titration) (see Table 3 for weight of adduct used and yields of products).

(b) *In benzene.* The adduct (1 mol.) was stirred in benzene (*ca.* 200 ml.) for 12 hr. Tetrahalogenodibenzamidetin(IV), tin tetrahalide, phenyl cyanide, and benzoic anhydride were formed and were separated and identified as described above. (See Table 3 for weight of adduct used and yields of products).

Reaction of Tin Tetrabromide (1·0 *mol.*) *with* NN-*Dibenzoylamine* (1·0 *mol.*).—Solid *NN*-dibenzoylamine (2·1 g., (1·0 mol.) was slowly added to a solution of tin tetrabromide (4·1 g., 1·0 mol.) in benzene (*ca.* 150 ml.), with stirring. After 4 hr. white *tetrabromo*(NN-*dibenzoylamine*)*tin*(IV) (6·0 g., 97%) (Found: C, 25·9; H, 1·8; Br, 48·0; N, 1·7; Sn, 17·7. $C_{14}H_{11}Br_4NO_2Sn$ requires C, 25·3; H, 1·7; Br, 48·2; N, 2·1; Sn, 17·9%) was filtered off and dried at 20°/1 mm. The complex sublimed quantitatively at 140°/1 mm.

Spectroscopy.—I.r. spectra were recorded (4000—400 cm^{-1}) with Perkin-Elmer 137 and 237 spectrophotomers and (450—80 cm^{-1}) with a Research and Industrial Instruments Company Ltd. FS-520 Michelson interferometer. Raman spectra of powdered solids were recorded using a Spex Raman spectrometer with 488·0 nm. Ar^+ laser excitation. The Mössbauer spectra were recorded as described previously.[15]

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