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# A tetranuclear $\beta$-diketonato complex of organotin(IV): synthesis and crystal structure of (benzoyltrifluoroacetonato)-(trifluoroacetato)tetramethyldistannoxane dimer $[\{C_6H_5C(\overline{O)CHCO(CF_3)S}n(CH_3)_2\}O\{Sn(CH_3)_2O_2CCF_3\}]_2$

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## Abstract

Condensation of dimethyltin oxide with $C_6H_5COCH_2COCF_3$ and $CF_3COOH$ in a 2 : 1 : 1 molar ratio in refluxing benzene yields the title compound which has been characterized by elemental analysis, multinuclear NMR spectroscopy, and an X-ray diffraction study. The structure contains chelating $\beta$-diketonate and monodentate trifluoroacetate moieties. The four-membered Sn–O–Sn–O ring formed as a result of dimerization is almost coplanar with the chelate ring. The coordination around both the tin atoms is distorted trigonal-bipyramidal, with the two carbons occupying equatorial positions with C–Sn–C bond angles of 143.1° and 135.9° respectively.

## 1. Introduction

Since the full characterization [1] of dimeric 1,3-dihalotetrabutylstannoxanes in 1961, the synthesis and structural chemistry of functionally substituted tetraorganodistannoxanes, both symmetrical as well as unsymmetrical, $XR_2SnOSnR_2X$ and $XR_2SnOSnR_2Y$, have received much attention and interest in the field continues [2,3]. These compounds, which invariably form dimers, find useful applications e.g. as catalysts in urethane formation [4]. In addition to detailed spectroscopic studies, both of the solid state and solutions (e.g. Mossbauer, IR, $^1H$ and $^{119}Sn$ NMR, *etc.*), the crystal structures of a number of these distannoxanes have been determined [2,3,5–13] and these show interesting structural variations depending upon the nature of the functional groups.

In view of this, the study of the distannoxanes containing bidentate chelating functional groups assumes significance. The chelation may influence the structure of the dimeric distannoxane strongly, and with suitable substituents on tin may give rise to a monomeric derivative. Reports so far on distannoxanes containing bidentate substituents have been concerned with carboxylato and substituted carboxylato derivatives only, and in all these cases the carboxylate moiety prefers to behave as a bridging group; functionally-substituted tetra-organodistannoxanes containing chelating moieties have not yet been characterized. The reaction of 1,3-diketones with 1,3-dichlorotetrabutyldistannoxane has been reported to cleave the Sn–O–Sn bond to yield the dibutyltin bis(1,3-diketone) derivative [14].

We report here the synthesis, spectroscopic characterization, and crystal structure of the first example of an unsymmetrical $\beta$-diketonato substituted tetraalkyldistannoxane containing a monodentate carboxylato group.

## 2. Results and discussion

Treatment of $Me_2SnO$ with $C_6H_5COCH_2COCF_3$ and $CF_3COOH$ in a 2 : 1 : 1 molar ratio in refluxing benzene solution gave the title compound. Surprisingly, the attempted synthesis of $Me_3SnOC(CF_3)CHCOC_6H_5$, by the reaction of $Me_3SnCl$ and $Na^+[COC(CF_3)CHCOC_6H_5]^-$ in 1 : 1 molar ratio in benzene also yielded the title compound after the removal of NaCl

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and the excess of solvent; the product could be purified by recrystallization from the benzene. The mechanism by which the product has been formed is uncertain but the incorporation of the trifluoroacetato group suggests that $CF_3COOH$ is an active participant, and it may be responsible for the cleavage of a methyl group from the tin atom. It is known that nucleophiles such as $OH^-$ will cleave β-diketones, to give in this case $CF_3COOH$ and $CH_3COC_6H_5$ [15]. Although in our case the reaction was carried out under anhydrous conditions, the ligand source $[CF_3COCHCOC_6H_5]^-Na^+$ may well have been hydrated making available water, which would be responsible for the cleavage of β-diketone. Moreover water must at some point enter the reaction scheme to generate the Sn–O–Sn moiety.

### 2.1. $^{19}F$ and $^{119}Sn$ NMR spectra

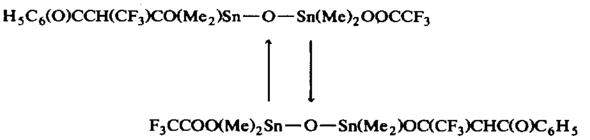
It is interesting that the $^{119}Sn$ NMR spectrum of the distannoxane shows only one signal at −122.7 ppm for both the tin atoms (along with a small signal at −328.7 ppm which corresponds to dimethyltin bis (β-diketonate)). The chemical shift indicates that the five-coordinate environment is retained at both the tin atoms even in solution. As indicated by the X-ray crystal structure, the two tin atoms have very similar environments which may explain the observation of only one signal in $CDCl_3$ solution. The signal is, however, slightly broad.

Variable-temperature $^{19}F$ NMR data reveal the tendency of the dimer to dissociate when heated in benzene. The spectrum at ambient temperature (20°C) shows a pair of signals at −79.7 and −74.1 ppm, along with another pair of very low intensity at −74.3 and −75.0 ppm. As the temperature is increased the intensity of the latter pair increases at the expense of the former, and finally at 70°C the intensity of the former pair becomes negligible.

The individual assignment of the above peaks for the trifluoroacetate and benzoyltrifluoroacetonate fluorines is very difficult because of the proximity of the signals. It may be mentioned here that the $^{19}F$ shift of trifluorobenzoyl acetone is −74.7 ppm and the reported [16] shift for various organotin trifluoroacetates are also in the same range (−74 to −79 ppm).

The molecular structure of the compound is shown in Fig. 1 (PLUTO) [17] along with the atom numbering scheme. Positional parameters of the non-H atoms and the equivalent values of the anisotropic temperature factors are given in Table 1 and the bond lengths and bond angles in Table 2. The structure reveals that there is the usual association found in distannoxane [2,3,5–13] involving oxygen bridges, and the unit cell contains dimeric molecules with crystallographically imposed $\bar{1}$ symmetry.

Linking of Sn(2) with stannoxane oxygen O(1) through a coordinate bond (the oxygen atom becomes three-coordinated) leads to a tetranuclear centrosymmetric dimer with planar central four-membered $Sn_2O_2$ ring:



$$\begin{array}{l} H_5C_6(O)CCH(CF_3)CO(Me_2)Sn—O—Sn(Me)_2OOCCF_3 \\ \qquad\qquad\qquad\qquad\qquad\qquad \uparrow\ \downarrow \\ \qquad\qquad\qquad F_3CCOO(Me)_2Sn—O—Sn(Me_2)OC(CF_3)CHC(O)C_6H_5 \end{array}$$

The distannoxane oxygen, O(1), forms two chemically equivalent bonds 2.028(4) and 2.049(4) Å with

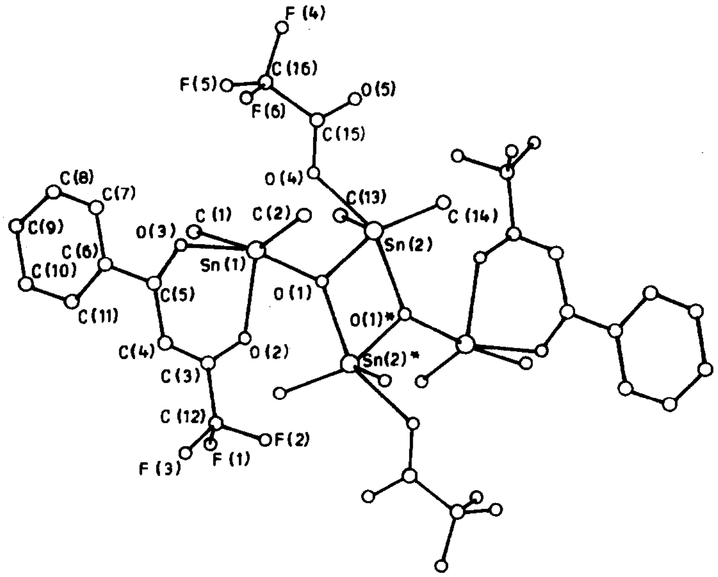


Fig. 1. Molecular structure of $[\{C_6H_5C(\overline{O)CHCO(CF_3)S}n(CH_3)_2\}O\{(CH_3)_2SnO_2CCF_3\}]_2$ showing the atomic numbering scheme.

TABLE 1. Positional parameters and their estimated standard deviations

| Atom | $x$ | $y$ | $z$ | $B$(Å$^2$) |
|---|---|---|---|---|
| Sn(1) | −0.24762(4) | 0.06176(2) | 0.64187(4) | 4.724(8) |
| Sn(2) | 0.02840(4) | 0.08349(2) | 0.47151(3) | 4.205(7) |
| F(1) | −0.3561(7) | −0.1845(2) | 0.6336(4) | 12.0(2) |
| F(2) | −0.2268(6) | −0.1791(2) | 0.7734(6) | 11.9(2) |
| F(3) | −0.4193(6) | −0.1946(2) | 0.7934(5) | 13.2(1) |
| F(4) | −0.1869(7) | 0.3411(2) | 0.5605(5) | 12.2(2) |
| F(5) | −0.3216(6) | 0.2730(4) | 0.4765(7) | 15.0(2) |
| F(6) | −0.2704(6) | 0.2551(3) | 0.6380(5) | 13.8(2) |
| O(1) | −0.0974(4) | 0.0215(2) | 0.5536(3) | 4.64(8). |
| O(2) | −0.2788(4) | −0.0475(2) | 0.6781(4) | 6.10(9) |
| O(3) | −0.4263(5) | 0.0681(2) | 0.7577(5) | 9.3(1) |
| O(4) | −0.1176(4) | 0.1625(2) | 0.5386(3) | 5.20(9) |
| O(5) | −0.0157(6) | 0.2548(3) | 0.4708(6) | 10.4(2) |
| C(1) | −0.3977(7) | 0.0876(4) | 0.5159(8) | 7.8(2) |
| C(2) | −0.1322(9) | 0.1037(4) | 0.7787(6) | 7.4(2) |
| C(3) | −0.3617(6) | −0.0790(3) | 0.7340(5) | 4.7(1) |
| C(4) | −0.4623(7) | −0.0522(3) | 0.7924(5) | 5.4(1) |
| C(5) | −0.4919(6) | 0.0218(3) | 0.8017(5) | 5.1(1) |
| C(6) | −0.6073(6) | 0.0460(3) | 0.8650(4) | 4.4(1) |
| C(7) | −0.6297(6) | 0.1191(3) | 0.8757(6) | 6.3(1) |
| C(8) | −0.7374(7) | 0.1429(4) | 0.9273(6) | 7.2(2) |
| C(9) | −0.8292(7) | 0.0957(4) | 0.9695(6) | 6.3(2) |
| C(10) | −0.8060(7) | 0.0244(4) | 0.9606(6) | 6.7(2) |
| C(11) | −0.6942(6) | −0.0005(3) | 0.9082(5) | 5.2(1) |
| C(12) | −0.3425(7) | −0.1594(3) | 0.7332(7) | 6.9(2) |
| C(13) | −0.0514(8) | 0.1050(5) | 0.3090(6) | 7.5(2) |
| C(14) | 0.1936(7) | 0.1212(4) | 0.5773(6) | 6.5(2) |
| C(15) | −0.1056(6) | 0.2283(3) | 0.5163(6) | 5.8(1) |
| C(16) | −0.2179(8) | 0.2746(4) | 0.5510(7) | 7.4(2) |

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2B_{1,1} + b^2B_{2,2} + c^2B_{3,3} + ab(\cos\gamma)B_{1,2} + ac(\cos\beta)B_{1,3} + bc(\cos\alpha)B_{2,3}]$.

Sn(2) and Sn(1) respectively and slightly longer bond with Sn(2)*, 2.115(4) Å. These bond distances are in close agreement with those reported [5] for the centrosymmetric dimeric stannoxane derivative, $[\{(n\text{-}C_4H_9)_2SnOOCCl_3\}_2O]$. The angles (O(1)–Sn(2)–O(1)) subtended at Sn(2) in the $Sn_2O_2$ four membered ring, are quite acute (75.8(2)°), with more open angles of 104.2(2)° at oxygen (Sn(2)–O(1)–Sn(2)*). These values are comparable with those of 75° and 105° found [5] in $[\{(n\text{-}C_4H_9)_2SnOOCCl_3\}_2O]$.

The environment around each tin atom appears to be best described as a distorted trigonal bipyramidal ($SnC_2O_3$) arrangement. Apical positions, as expected, are occupied by oxygen atoms. Two carbon atoms (of methyl groups) and one oxygen atom are in the equatorial plane. The angular distortion in the planes seems to be due to the differences in electronegativity of the attached groups. In fact, the methyl groups subtend angles of 143.1(3)° and 135.9(4)° at Sn(1) and Sn(2) respectively while the mean methyl–tin–oxygen angles are 105.8° and 111.7(7)°. The sums of the angles in the equatorial plane at Sn(1) and Sn(2) is 354.7° and 329.2°. The bond angles between two axial oxygen atoms at Sn(1), (O(1)–Sn(1)–O(3), 161.1(9)°) and at Sn(2), (O(1)*–Sn(2)–O(4), 152.0(1)°) are less than usual (180°).

The trifluoroacetate group is attached to Sn(2) and is inclined at an angle of 8.67° to the central $Sn_2O_2$ skeleton. The trifluoroacetate group functions essentially as a unidentate ligand. The two C–O bond distances O(5)–C(15) of 1.194(9) Å and O(4)–C(15) of 1.273(7) Å are non-equivalent. The former reflects the

TABLE 2. Intramolecular distances (Å) and angles (°) with estimated standard deviations in parentheses

| | | | |
|---|---|---|---|
| *Bonds* | | | |
| Sn(1)–O(1) | 2.049(4) | O(2)–C(3) | 1.256(7) |
| Sn(1)–O(2) | 2.125(4) | O(3)–C(5) | 1.234(8) |
| Sn(1)–O(3) | 2.354(5) | O(4)–C(15) | 1.273(7) |
| Sn(1)–C(1) | 2.103(9) | O(5)–C(15) | 1.194(9) |
| Sn(1)–C(2) | 2.096(7) | C(3)–C(4) | 1.365(9) |
| Sn(2)–O(1) | 2.028(4) | C(3)–C(12) | 1.522(8) |
| Sn(2)–O(4) | 2.268(4) | C(4)–C(5) | 1.428(8) |
| Sn(2)–C(13) | 2.113(7) | C(5)–C(6) | 1.498(8) |
| Sn(2)–C(14) | 2.117(7) | C(6)–C(7) | 1.397(8) |
| F(1)–C(12) | 1.302(9) | C(6)–C(11) | 1.361(8) |
| F(2)–C(12) | 1.260(9) | C(7)–C(8) | 1.36(1) |
| F(3)–C(12) | 1.282(9) | C(8)–C(9) | 1.40(2) |
| F(4)–C(16) | 1.289(9) | C(9)–C(10) | 1.36(2) |
| F(5)–C(16) | 1.31(2) | C(10)–C(11) | 1.401(9) |
| F(6)–C(16) | 1.27(2) | C(15)–C(16) | 1.50(2) |
| | | Sn(2) * –O(1) | 2.115(4) |
| *Angles* | | | |
| O(1)–Sn(1)–O(2) | 82.9(2) | O(3)–C(5)–C(6) | 117.5(5) |
| O(1)–Sn(1)–O(3) | 161.1(1) | C(4)–C(5)–C(6) | 120.4(5) |
| O(1)–Sn(1)–C(1) | 101.4(3) | C(5)–C(6)–C(7) | 118.6(5) |
| O(1)–Sn(1)–C(2) | 100.9(3) | C(5)–C(6)–C(11) | 122.4(5) |
| O(2)–Sn(1)–O(3) | 78.2(2) | C(7)–C(6)–C(11) | 118.9(5) |
| O(2)–Sn(1)–C(1) | 105.7(2) | C(6)–C(7)–C(8) | 120.2(6) |
| O(2)–Sn(1)–C(2) | 105.9(2) | C(7)–C(8)–C(9) | 121.4(6) |
| O(3)–Sn(1)–C(1) | 84.9(3) | C(0)–C(9)–C(10) | 118.3(6) |
| O(3)–Sn(1)–C(2) | 83.5(3) | C(9)–C(10)–C(11) | 120.6(6) |
| C(1)–Sn(1)–C(2) | 143.1(3) | C(6)–C(11)–C(10) | 120.7(5) |
| O(1)–Sn(2)–O(4) | 76.3(1) | F(1)–C(12)–F(2) | 106.2(7) |
| O(1)–Sn(2)–C(13) | 112.2(2) | F(1)–C(12)–F(3) | 109.3(6) |
| O(1)–Sn(2)–C(14) | 111.3(2) | F(11)–C(12)–C(3) | 111.2(6) |
| O(4)–Sn(2)–C(13) | 90.9(2) | F(2)–C(12)–F(3) | 100.7(6) |
| O(4)–Sn(2)–C(14) | 92.4(2) | F(2)–C(12)–C(3) | 113.4(5) |
| C(13)–Sn(2)–C(14) | 135.9(4) | F(3)–C(12)–C(3) | 115.4(6) |
| Sn(1)–O(1)–Sn(2) | 123.2(2) | O(4)–C(15)–O(5) | 126.2(6) |
| Sn(1)–O(2)–C(3) | 132.8(4) | O(4)–C(15)–C(16) | 114.7(6) |
| Sn(1)–O(3)–C(5) | 132.3(5) | O(5)–C(15)–C(16) | 119.2(6) |
| Sn(2)–O(4)–C(15) | 118.9(4) | F(4)–C(16)–F(5) | 104.5(7) |
| O(2)–C(3)–C(4) | 130.2(6) | F(4)–C(16)–F(6) | 108.2(7) |
| O(2)–C(3)–C(12) | 112.1(5) | F(4)–C(16)–C(15) | 114.3(7) |
| C(4)–C(3)–C(12) | 117.7(5) | F(5)–C(16)–F(6) | 103.0(7) |
| C(3)–C(4)–C(5) | 124.2(6) | F(5)–C(16)–C(15) | 110.1(7) |
| O(3)–C(5)–C(4) | 122.3(6) | F(6)–C(16)–C(15) | 115.6(6) |
| Sn(2)–O(1)–Sn(2) * | 104.2(2) | O(1)–Sn(2)–O(1) * | 75.8(2) |
| Sn(1)–O(1)–Sn(2) * | 132.5(2) | | |

* Symmetry element is $-x$, $-y$, $1-z$.

presence of the free C=O group in the trifluoroacetate moiety. The Sn(2)–O(4) bond distance is similar to that reported [8] for $[\{(CH_2{=}CH)_4Sn_2(OOCCF_3)_2\}O]_2$. The two tin–carbon bonds, [Sn(2)–C(13), 2.113(7) Å and Sn(2)–C(14), 2.117(7) Å], are equivalent and normal.

It is noteworthy that although Sn(2) is chelated by the benzoyltrifluoroacetonate moiety and Sn(1) is bonded to the trifluoroacetate moiety, the geometries around both tin atoms are similar. The Sn(1)–O(2) equatorial and Sn(1)–O(3) axial distances are 2.125(4) Å and 2.354(5) Å respectively. The difference of 0.23 Å is quite significant, but the values are comparable to the reported [9] Sn–O bond distances in ($Ph_3SnONPh$-COPh), which contains both covalent (2.091 Å) and coordinate (2.308 Å) Sn–O interactions.

The weakening of the Sn(1)–O(3) bond may be related either to the *trans* effect of a very strong Sn(1)–O(1) axial 2.049(4) Å bond or to the steric requirements of the ligands. The contribution of the enolate form to the overall structure is manifested in the C–O and C–C interatomic distances within the benzoyltrifluoroacetone ligand. The C(5)–O(3) distance of 1.234(8) Å is slightly shorter than that of C(3)–O(2) (1.256(7) Å), but the C(3)–C(4) bond of length 1.365(9) Å, is appreciably shorter, and hence has more double bond character, than the corresponding C(4)–C(5) bond, of length 1.428(8) Å. However, both C–C distances are shorter than the two exocyclic C–C distances (1.508(8) and 1.497(8) Å). All these distances are similar to those reported [10] for $Sn(bzac)_2$. The two methyl groups attached to Sn(1), (Sn(1)–C(1), 2.103(9) Å and Sn(1)–C(2), 2.096(7) Å), are equivalent.

The chelate ring of the ligand is effectively coplanar with the $Sn_2O_2$ plane, making an angle of 2.12° with the latter. The phenyl ring attached to the ligand is almost coplanar with the plane of the ligand and the $Sn_2O_2$ skeleton, being inclined to these planes at angles of 7.72° and 6.24°, respectively.

## 3. Experimental details

All solvents were of analytical grade and moisture-free and were dried by standard methods. Dimethyltin oxide (Merck) was used as supplied. Trifluorobenzoylacetone and trifluoroacetic acid were distilled before use.

### 3.1. *Reaction of dimethyltin oxide with benzoyltrifluoroacetone and trifluoroacetic acid*

A benzene solution of benzoyltrifluoroacetone (0.83 g, 3.83 mmol) and trifluoroacetic acid (0.43 g, 3.78 mmol) was mixed with a benzene suspension of dimethyltin oxide (1.25 g, 7.57 mmol). The mixture was refluxed under a fractionating column. A small amount of water formed during the course of the reaction was removed azeotropically with benzene. After 2 h refluxing, a clear solution was obtained. The excess of solvent was distilled off and the residue was kept under reduced pressure to yield a lemon-yellow sticky solid, which was recrystallized from a benzene/n-hexane mixture.

TABLE 3. Crystallographic data for $C_{32}H_{36}O_{10}F_{12}Sn_4$

| | |
|---|---|
| Formula | $C_{32}H_{36}O_{10}F_{12}Sn_4$ |
| Mol. wt. | 1283.38 |
| $a$ (Å) | 9.870(4) |
| $b$ (Å) | 18.770(7) |
| $c$ (Å) | 12.214(3) |
| $\beta$ (°) | 94.75(2) |
| $V$ (Å$^3$) | 2255.3 Å$^3$ |
| $Z$ | 2 |
| Space group | $P2_{1/c}$ (No. 14) |
| $d_{calc.}$ (g cm$^{-3}$) | 1.89 |
| Mo-K$\alpha$ (cm$^{-1}$) | 9.1 |
| $F(000)$ | 1232 |
| Temp. (°C) | 22 |
| No. of parameters refined | 262 |
| $R(F_o)$ | 0.036 |
| $R_w(F_o)$ | 0.050 |

Anal. Found: Sn, 36.90; C, 29.95; H, 2.76. $C_{32}H_{36}O_{10}F_{12}Sn_4$ calcd.: Sn, 36.99; C, 29.92; H, 2.80%.

## 4. Crystal data

A light yellow crystal *ca.* 25 × 0.35 × 0.17 mm was mounted on a glass fibre using an epoxy resin on an Enraf–Nonius CAD-4 diffractometer. Relevant crystallographic details are given in Table 3. Accurate unit cell parameters were obtained by means of least-squares analyses of 25 reflections. Intensity data on the compound were then collected in the $\omega$-$2\theta$ scan mode with scan width of $0.7 + 0.35 \tan \theta$. A variable scan speed with a maximum scan time of one minute was used. Two standard reflections monitored (126 and 226) every 1800 seconds showed no significant variations over the data collection. There was no crystal decay. The intensities were corrected for Lorentz and polarization factors but not for absorption. All computations were performed on a Digital PDP11/73 computer.

The structure was solved by a routine heavy-atom method with SDP software [16] by a combination of Patterson, difference Fourier, and full-matrix-least-squares refinement techniques, using anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms were omitted. Out of 4098 unique reflections measured for $2° < \theta < 25°$ and $h$(0-11), $k$(0-22),

$\pm l(-14\text{-}14)$, 3221 with $|F^2| > \sigma(F^2)$ were used in the refinement where $\sigma(F^2) = \{\sigma^2(I) + (0.04I)^2\}^{1/2}/L_p$.

The final residuals were $R = 0.036$ and $R_w = 0.050$ where

$$R = \sum \| F_o | - | F_c \| / \sum | F_o |,$$

$$R_w = \left[ \sum w \| F_o | - | F_d |^2 / \sum w | F_o |^2 \right]^{1/2} \text{ and}$$

$$w = 1/\sigma^2(F_o)$$

The atomic scattering factors were taken from ref. 17.

List of thermal parameters and studied factors are available from the authors.

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