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> cis-Bis(acetonitrile)tetrachlorotin(IV) acetonitrile solvate, cis-tetrachlorobis(propiononitrile)tin(IV) propiononitrile solvate, cis-tetrachlorobis(isobutyronitrile)tin(IV), cis-tetrachlorobis(cyclohexanecarbonitrile)tin(IV) and cis-tetrachlorobis(o-toluonitrile)tin(IV), all determined at ca 150 K

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The structures of the title compounds, $\left[\mathrm{SnCl}_{4}\left(\mathrm{C}_{2} \mathrm{H}_{3} \mathrm{~N}\right)_{2}\right]$-$\mathrm{C}_{2} \mathrm{H}_{3} \mathrm{~N}, \quad\left[\mathrm{SnCl}_{4}\left(\mathrm{C}_{3} \mathrm{H}_{5} \mathrm{~N}\right)_{2}\right] \cdot \mathrm{C}_{3} \mathrm{H}_{5} \mathrm{~N}, \quad\left[\mathrm{SnCl}_{4}\left(\mathrm{C}_{4} \mathrm{H}_{7} \mathrm{~N}\right)_{2}\right],\left[\mathrm{SnCl}_{4}-\right.$ $\left.\left(\mathrm{C}_{7} \mathrm{H}_{11} \mathrm{~N}\right)_{2}\right]$ and $\left[\mathrm{SnCl}_{4}\left(\mathrm{C}_{8} \mathrm{H}_{7} \mathrm{~N}\right)_{2}\right]$, were determined with the intention of examining the effect of various substituent types in nitrile ligands, $R C N$, behaving in a common $\sigma$-donor situation \{in this case, as cis-bis complexes with $\mathrm{SnCl}_{4}$, viz. $\left.\left[\mathrm{SnCl}_{4}(\mathrm{RCN})_{2}\right]\right\}$, on (i) the strength of complex formation with the metal atom and (ii) other bonding behaviour of the metal (for example, trans effects). The five structures exhibit no nontrivial systematic perturbation that can be said to be contingent on the substituent type.

## Comment

Organic nitrile ligands in their coordination complexes with metal atoms are commonly regarded as both $\sigma$-donors and $\pi$-donors, which, having a triple bond ( $R \mathrm{C} \equiv \mathrm{N}$ ), should be particularly appropriate for the transmission of electronic effects of the $R$ substituent through the bonding scheme linking it to the metal. Presumably, any such effect will be superimposed on the common observation of the lengthening of the $\mathrm{C} \equiv \mathrm{N}$ bond on coordination. The substituent is well removed from the metal and should not interact sterically with the rest of the coordination environment, unless the substituent is unusually bulky. However, the rod-like nature of the ligand may render it susceptible to substantial consequences of packing forces, which are usually manifested in deviations from linearity of the $M N C$ linkage, $N C R$ being less susceptible. Such effects can be explored spectroscopically, but in the
present context, we wanted to examine the question from a structural perspective. The chosen vehicle for the study was the array of bis(monodentate nitrile) complexes of stannic chloride. Only the structure of the acetonitrile complex has been recorded previously (Webster \& Blayden, 1969), showing the metal-atom stereochemistry to be quasi-octahedral sixcoordinate, with the nitrile ligands in cis positions in the coordination sphere, cis- $\left[\mathrm{SnCl}_{4}(\mathrm{MeCN})_{2}\right]$, in a situation where the $\sigma$-donor character of the ligand might be expected to predominate. [Two determinations of the one-dimensional polymer stannic chloride-glutaronitrile (1/1) are recorded (Barnhart et al., 1968; Liu, 1985), also with two nitrile donors from different ligands in cis positions in the coordination sphere.]

Attempts were made to crystallize a number of such complexes from solutions of stannic chloride in various nitriles under Schlenk conditions, by volume reduction and/or by cooling as necessary. Well formed colourless crystals were obtained for a diverse array of substituents, $R[R$ is methyl, ethyl, isopropyl, cyclohexyl (cy) and $o$-tolyl ( $o$-tol)], and were subjected to structural study.

Stannic chloride crystallizes from acetonitrile solution as $\mathrm{SnCl}_{4} \cdot 3 \mathrm{MeCN}$. In the belief that one of the acetonitrile groups was uncoordinated in the original determination, $\mathrm{SnCl}_{4} \cdot-$ 2 MeCN was obtained by pumping on the solid in vacuo and recrystallizing the material from carbon tetrachloride, the results of the determination establishing the stereochemistry (Webster \& Blayden, 1969). In the present study, it was considered worth establishing the nature of the 3 MeCN adduct, which was found to be the monosolvate of the cis-bis complex, cis-[ $\left.\mathrm{SnCl}_{4}(\mathrm{MeCN})_{2}\right] \cdot \mathrm{MeCN}$, (I). The asymmetric unit of the structure contains one-half of the formula unit. The complex molecule is disposed about the crystallographic mirror plane in space group Pnma, and the Sn atom and the pair of mutually trans Cl atoms lie in the plane. The substrate molecule is well defined (Fig. 1a), but not so much as to permit refinement of the H -atom parameters at the periphery of the ligand, where the displacement parameters are higher. The MeCN molecule of crystallization is more problematic; it is seemingly disordered over a continuous undulating sequence of residues passing through the cell in the $b$ direction and is modelled with the terminal C atom in the mirror plane, with full site occupancy, and the CN group disposed to either side, with an occupancy of 0.5 (Fig. 1b).


The propiononitrile adduct, similarly a cis-bis(ligand) complex, is also a monosolvate, viz. cis- $\left[\mathrm{SnCl}_{4}(\mathrm{EtCN})_{2}\right] \cdot \mathrm{EtCN}$,
(II), and there is one formula unit devoid of crystallographic symmetry in the asymmetric unit of the structure. A projection of the molecule is shown in Fig. 2(a); the molecules stack in columns along $b$, with the voids between them occupied by the uncoordinated solvent molecules (Fig. 2b). The cyclohexylcarbonitrile (cyCN) adduct, (IV), is also of the same type, viz. it is unsolvated and there is one molecule of cis-[ $\mathrm{SnCl}_{4}$ $(\mathrm{cyCN})_{2}$ ], devoid of crystallographic symmetry, in the asymmetric unit (Fig. 3a); the cyclohexyl rings adopt 'chair' configurations, with the associated CN substituents appended axially.

The $o$-toluonitrile $\left(o\right.$-tolCN) adduct, $c i s$ - $\left[\mathrm{SnCl}_{4}(o \text {-tolCN })_{2}\right]$, (V), is likewise unsolvated, with one molecule (Fig. 3b) in the asymmetric unit. The similarly unsolvated isobutyronitrile adduct, $\left[\mathrm{SnCl}_{4}\left({ }^{\mathrm{i}} \mathrm{PrCN}\right)_{2}\right]$, (III), has two molecules devoid of crystallographic symmetry in the asymmetric unit. Geometric

(a)

(b)

Figure 1
(a) A molecular projection of the cis- $\left[\mathrm{SnCl}_{4}(\mathrm{MeCN})_{2}\right]$ part of (I). Displacement ellipsoids are shown at the $50 \%$ probability level for non-H atoms. (b) A projection of the unit cell of (I) along $c$. The crystallographic mirror plane passing through the molecule lies at $y=\frac{1}{4}$ etc. The strings of solvate residues passing through the structure in the $b$ direction are shown.
parameters of the individual molecules are summarized in Tables 1-5 and the molecules are depicted in Figs. 1-3.

The earlier structure of $\left[\mathrm{SnCl}_{4}(\mathrm{MeCN})_{2}\right]$ (Webster \& Blayden, 1969) was derived from a room-temperature film determination and, although not inharmonious with the present determination, is not sufficiently precise to justify inclusion in any survey of trends or correlations. In general, the molecules of all five title complexes are of the form cis[(nitrile ligand) ${ }_{2} \mathrm{SnCl}_{4}$ ], with or without an uncoordinated solvent molecule. Defining the trans $\mathrm{Cl}-\mathrm{Sn}-\mathrm{Cl}$ group as 'axial' and the remaining $\mathrm{N}_{2} \mathrm{SnCl}_{2}$ quasi-plane as 'equatorial' (eq), we find in all cases that the $\mathrm{Cl}_{\mathrm{eq}}-\mathrm{Sn}-\mathrm{Cl}_{\mathrm{eq}}$ angle is enlarged beyond $90^{\circ}$ by a significant margin, while the $\mathrm{N}-$ $\mathrm{Sn}-\mathrm{N}$ angle is concomitantly diminished. This behaviour is consistent with the $\mathrm{Sn}-\mathrm{N}$ bonds being weaker than the $\mathrm{Sn}-$ Cl bonds. The $\mathrm{Cl}_{\mathrm{ax}}-\mathrm{Sn}-\mathrm{Cl}_{\mathrm{ax}}$ angle deviates significantly from linearity, being folded towards the N atoms and away from the 'equatorial' Cl atoms, which again is consistent with the bonds from the latter being stronger (Kepert, 1982).

Focusing on possible correlations between stereochemical parameters and substituents is little more enlightening. The $\mathrm{Sn}-\mathrm{Cl}_{\mathrm{ax}}$ and $\mathrm{Sn}-\mathrm{Cl}_{\text {eq }}$ bonds in all five compounds fall in a


Figure 2
(a) A molecular projection of the cis- $\left[\mathrm{SnCl}_{4}(\mathrm{EtCN})_{2}\right]$ part of (II). Displacement ellipsoids are shown at the $50 \%$ probability level for non-H atoms. (b) A projection of the unit cell of (II) along $b$, showing the tunnels through the structure containing the solvent molecules.

(a)

(c)

(b)

(b)

Figure 3
 (III). Displacement ellipsoids are shown at the $50 \%$ probability level for non-H atoms.
narrow range $[2.3520(11)-2.3761(12) ~ \AA$ ] , with relatively wide divergences in any given bond type for a specific compound [e.g. the $\mathrm{Sn}-\mathrm{Cl}_{\mathrm{ax}}$ bonds for $R=\mathrm{cy}$ are 2.3488 (5) and 2.3732 (5) Å]. The $\mathrm{Sn}-\mathrm{N}$ distances range between 2.255 (4) and 2.276 (4) $\AA$, again with disparities within the familial groups (both of these values are for the isobutyronitrile adduct), rendering attempts to extract significant trends from what is again a narrow range thoroughly insecure. The same is true of the $\mathrm{N}-\mathrm{C}$ distances $[1.126$ (6)-1.1428 (18) $\AA$ ]. These values are similar to those reported recently for a free acetonitrile ligand [1.141 (2) Å; Brackemeyer et al., 1997], which implies that the numerous suggestions of a dependence of this bond on coordination, admittedly contingent on the nature of the metal, perhaps should be subject to closer scrutiny and that libration corrections may be necessary. The range of $\mathrm{Sn}-\mathrm{N}-\mathrm{C}$ angles found in single compounds [e.g. 158.21 (15)-167.85 (15) ${ }^{\circ}$ for $R=o$-tol and 163.2 (4)-174.8 (3) ${ }^{\circ}$ for $R={ }^{i} \mathrm{Pr}$ ] suggest that 'packing forces' may be responsible for the many substantial perturbations and variations evident in the other angles. The above results suggest that substituent effects in nitrile ligand complexes of the type presented here
may be difficult to detect and apprehend by structural methods alone and that spectroscopic methods (e.g. Kawano et al., 1976) may be more apposite when based on precise geometries, such as those offered here. Finally, note that, although the cis configuration of the above compounds is consistent with weak $\mathrm{Sn}-\mathrm{N}$ bonds (as supported by bondangle evidence), the $\mathrm{Sn}-\mathrm{N}$ and $\mathrm{Sn}-\mathrm{Cl}$ distances are little different from those in $\left[\mathrm{SnCl}_{4}(\mathrm{bpy})\right]$ [bpy is bipyridyl; $\mathrm{Sn}-\mathrm{N}=2.226$ (4) and 2.247 (4) $\AA$, and $\mathrm{Sn}-\mathrm{Cl}=2.359$ (2)2.409 (1) $\AA$; Zakharov et al., 1991] and $\left[\mathrm{SnCl}_{4}(\right.$ phen $\left.)\right] \cdot \mathrm{C}_{6} \mathrm{H}_{6}$ [phen is 1,10 -phenanthroline; $\mathrm{Sn}-\mathrm{N}=2.234$ (8)-2.251 (8) $\AA$ and $\mathrm{Sn}-\mathrm{Cl}=2.361$ (3)-2.410 (4) $\AA$; Hall \& Tiekink, 1996].

## Experimental

All compounds, being highly sensitive to exposure to air, were obtained by the addition of distilled stannic chloride to the appropriate nitrile ( $\sim 10 \mathrm{ml}$, freshly distilled from calcium hydride) in a Schlenk tube. The reaction mixtures were allowed to stand until crystals were deposited, and these crystals were transferred directly from the Schlenk tube to the low-temperature stream.

## Compound (I)

## Crystal data

$\left[\mathrm{SnCl}_{4}\left(\mathrm{C}_{2} \mathrm{H}_{3} \mathrm{~N}\right)_{2}\right] \cdot \mathrm{C}_{2} \mathrm{H}_{3} \mathrm{~N}$
$M_{r}=383.68$
Orthorhombic, Pnma
$a=10.480$ (2) A
$b=13.783$ (2) $\AA$
$c=9.758$ (2) A
$V=1409.5$ (4) $\AA^{3}$
$Z=4$
$D_{x}=1.808 \mathrm{Mg} \mathrm{m}^{-3}$

Data collection
Bruker SMART CCD
diffractometer
$\omega$ scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{\text {min }}=0.64, T_{\text {max }}=0.80$
13399 measured reflections

## Refinement

Refinement on $F$
$R=0.019$
$w R=0.022$
$S=1.16$
1726 reflections
80 parameters
H -atom parameters not refined
$w=1 /\left(\sigma^{2} F+0.002 F^{2}\right)$

Table 1
Selected geometric parameters $\left(\AA^{\circ},{ }^{\circ}\right)$ for (I).

|  |  |  |  |
| :--- | :--- | :--- | :--- |
| $\mathrm{Sn}-\mathrm{Cl} 1$ | $2.3518(10)$ | $\mathrm{N} 11-\mathrm{C} 11$ | $1.132(3)$ |
| $\mathrm{Sn}-\mathrm{Cl} 2$ | $2.3641(10)$ | $\mathrm{C} 11-\mathrm{C} 12$ | $1.453(4)$ |
| $\mathrm{Sn}-\mathrm{Cl} 3$ | $2.3635(7)$ | $\mathrm{N} 21-\mathrm{C} 21$ | $1.137(18)$ |
| $\mathrm{Sn}-\mathrm{N} 11$ | $2.259(2)$ | $\mathrm{C} 21-\mathrm{C} 22$ | $1.605(15)$ |
|  |  |  |  |
|  |  |  | $172.56(5)$ |
| $\mathrm{Cl} 1-\mathrm{Sn}-\mathrm{Cl} 2$ | $164.87(3)$ | $\mathrm{Cl} 3-\mathrm{Sn}-\mathrm{N} 11$ | $178.5(2)$ |
| $\mathrm{Cl} 1-\mathrm{Sn}-\mathrm{Cl} 3$ | $95.77(2)$ | $\mathrm{Sn}-\mathrm{N} 11-\mathrm{C} 11$ | $178.9(3)$ |
| $\mathrm{Cl} 1-\mathrm{Sn}-\mathrm{N} 11$ | $85.03(6)$ | $\mathrm{N} 11-\mathrm{C} 11-\mathrm{C} 12$ | $178.5(10)$ |
| $\mathrm{Cl} 2-\mathrm{Sn}-\mathrm{Cl} 3$ | $94.12(2)$ | $\mathrm{N} 21-\mathrm{C} 21-\mathrm{C} 22$ |  |
| $\mathrm{Cl} 2-\mathrm{Sn}-\mathrm{N} 11$ | $83.71(6)$ |  |  |
|  |  |  |  |

## Compound (II)

## Crystal data

Cell parameters from 8148 reflections
$\theta=2.6-26^{\circ}$
$\mu=2.54 \mathrm{~mm}^{-1}$
$T=150$ (2) K
Prism, colourless
$0.24 \times 0.20 \times 0.15 \mathrm{~mm}$

1841 independent reflections
1726 reflections with $I>2 \sigma(I)$
$R_{\text {int }}=0.025$
$\theta_{\text {max }}=28.4^{\circ}$
$h=-13 \rightarrow 13$
$k=-18 \rightarrow 18$
$l=-13 \rightarrow 13$
$(\Delta / \sigma)_{\max }=0.024$
$\Delta \rho_{\text {max }}=0.57 \mathrm{e}^{\AA^{-3}}$
$\Delta \rho_{\text {min }}=-0.54 \mathrm{e}^{-3}$
Extinction correction: Zachariasen (1967)

Extinction coefficient:
$1.07(5) \times 10^{3}$

## Refinement

Refinement on $F$
H -atom parameters not refined
$R=0.027$
$w=1 /\left(\sigma^{2} F+0.0004 F^{2}\right)$
$(\Delta / \sigma)_{\text {max }}=0.008$
$w R=0.034$
$\Delta \rho_{\text {max }}=0.93 \mathrm{e}^{-3}$
$\Delta \rho_{\text {min }}=-0.76 \mathrm{e}^{-3}$
$S=1.11$
4991 reflections
154 parameters
$\left[\mathrm{SnCl}_{4}\left(\mathrm{C}_{3} \mathrm{H}_{5} \mathrm{~N}\right)_{2}\right] \cdot \mathrm{C}_{3} \mathrm{H}_{5} \mathrm{~N}$
$M_{r}=425.76$
Orthorhombic, $P$ Pbca
$a=11.274(2) \AA$
$b=13.822(2) \AA$
$c=21.606(3) \AA$
$V=336.8(9) \AA^{3}$
$Z=8$
$D_{x}=1.680 \mathrm{Mg} \mathrm{m}^{-3}$

## Data collection

| Bruker SMART CCD | 6697 independent reflections |
| :--- | :--- |
| $\quad$ diffractometer | 4991 reflections with $I>2 \sigma(I)$ |
| $\omega$ scans | $R_{\text {int }}=0.042$ |
| Absorption correction: multi-scan | $\theta_{\max }=34.1^{\circ}$ |
| $\quad(S A D A B S ;$ Sheldrick, 1996) | $h=-17 \rightarrow 17$ |
| $T_{\min }=0.43, T_{\max }=0.75$ | $k=-21 \rightarrow 21$ |
| 50732 measured reflections | $l=-34 \rightarrow 34$ |

50732 measured reflections
Mo $K \alpha$ radiation
Cell parameters from 6924 reflections
$\theta=2.6-29.3^{\circ}$
$\mu=2.14 \mathrm{~mm}^{-1}$
$T=150(2) \mathrm{K}$
Block, colourless
$0.40 \times 0.20 \times 0.15 \mathrm{~mm}$
m364

Data collection

## Bruker SMART CCD

 diffractometer$\omega$ scans
Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)
$T_{\min }=0.67, T_{\max }=0.83$
30610 measured reflections

## Refinement

Refinement on $F$
$R=0.033$
$w R=0.04$
$S=1.23$
5976 reflections
271 parameters

## Compound (IV)

## Crystal data

$\left[\mathrm{SnCl}_{4}\left(\mathrm{C}_{7} \mathrm{H}_{11} \mathrm{~N}\right)_{2}\right]$
$M_{r}=478.87$
Monoclinic, $P 2_{1} / c$
$a=10.0990$ (15) $\AA$
$b=15.772$ (2) $\AA$
$c=12.5100(19) \AA$
$\beta=104.719(3)^{\circ}$
$V=1927.2(5) \AA^{3}$
$Z=4$
7872 independent reflections
5976 reflections with $I>2 \sigma(I)$
$R_{\text {int }}=0.041$
$\theta_{\text {max }}=29$.
$h=-21 \rightarrow 20$
$k=-16 \rightarrow 16$
$l=-22 \rightarrow 22$

H -atom parameters not refined $w=1 /\left(\sigma^{2} F+0.0004 F^{2}\right)$
$(\Delta / \sigma)_{\max }=0.003$
$\Delta \rho_{\max }=1.74 \mathrm{e}^{-3}$
$\Delta \rho_{\text {min }}=-0.97 \mathrm{e}^{-3}$

Data collection
Bruker SMART CCD diffractometer
$\omega$ scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{\text {min }}=0.49, T_{\text {max }}=0.75$
28748 measured reflections

## Refinement

Refinement on $F$
$R=0.021$
$w R=0.03$
$S=1.10$
6663 reflections
278 parameters

## Data collection

Bruker SMART CCD
diffractometer

## $\omega$ scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)
$T_{\text {min }}=0.72, T_{\text {max }}=0.77$
29755 measured reflections

## Refinement

Refinement on $F$
$R=0.027$
$w R=0.034$
$S=0.99$
6555 reflections
208 parameters

7717 independent reflections 6555 reflections with $I>2 \sigma(I)$
$R_{\text {int }}=0.031$
$\theta_{\text {max }}=34.0^{\circ}$
$h=-23 \rightarrow 22$
$k=-11 \rightarrow 11$
$l=-28 \rightarrow 28$

Table 5
Selected geometric parameters $\left(\AA^{\circ}{ }^{\circ}\right)$ for (V).

| $\mathrm{Sn}-\mathrm{Cl} 1$ | $2.3560(7)$ | $\mathrm{Sn}-\mathrm{N} 1$ | $2.2687(16)$ |
| :--- | :---: | :--- | :---: |
| $\mathrm{Sn}-\mathrm{Cl} 2$ | $2.3678(6)$ | $\mathrm{Sn}-\mathrm{N} 2$ | $2.2658(16)$ |
| $\mathrm{Sn}-\mathrm{Cl} 3$ | $2.3606(7)$ | $\mathrm{N} 1-\mathrm{C} 1$ | $1.139(2)$ |
| $\mathrm{Sn}-\mathrm{Cl} 4$ | $2.3488(7)$ | $\mathrm{N} 2-\mathrm{C} 2$ | $1.142(2)$ |
|  |  |  |  |
|  |  |  | $172.36(4)$ |
| $\mathrm{Cl} 1-\mathrm{Sn}-\mathrm{Cl} 2$ | $95.67(2)$ | $\mathrm{Cl} 3-\mathrm{Sn}-\mathrm{N} 1$ | $88.92(4)$ |
| $\mathrm{Cl} 1-\mathrm{Sn}-\mathrm{Cl} 3$ | $94.78(2)$ | $\mathrm{Cl} 3-\mathrm{Sn}-\mathrm{N} 2$ | $84.02(5)$ |
| $\mathrm{Cl} 1-\mathrm{Sn}-\mathrm{Cl} 4$ | $163.987(19)$ | $\mathrm{Cl} 4-\mathrm{Sn}-\mathrm{N} 1$ | $84.28(5)$ |
| $\mathrm{Cl} 1-\mathrm{Sn}-\mathrm{N} 1$ | $84.00(5)$ | $\mathrm{Cl} 4-\mathrm{Sn}-\mathrm{N} 2$ | $83.45(6)$ |
| $\mathrm{Cl} 1-\mathrm{Sn}-\mathrm{N} 2$ | $83.84(5)$ | $\mathrm{N} 1-\mathrm{Sn}-\mathrm{N} 2$ | $167.85(15)$ |
| $\mathrm{Cl} 2-\mathrm{Sn}-\mathrm{Cl} 3$ | $98.13(2)$ | $\mathrm{Sn}-\mathrm{N} 1-\mathrm{C} 1$ | $178.6(2)$ |
| $\mathrm{Cl} 2-\mathrm{Sn}-\mathrm{Cl} 4$ | $94.82(2)$ | $\mathrm{N} 1-\mathrm{C} 1-\mathrm{C} 11$ | $158.21(15)$ |
| $\mathrm{Cl} 2-\mathrm{Sn}-\mathrm{N} 1$ | $89.50(4)$ | $\mathrm{Sn}-\mathrm{N} 2-\mathrm{C} 2$ | $178.5(2)$ |
| $\mathrm{Cl} 2-\mathrm{Sn}-\mathrm{N} 2$ | $172.95(4)$ | $\mathrm{N} 2-\mathrm{C} 2-\mathrm{C} 21$ |  |
| $\mathrm{Cl} 3-\mathrm{Sn}-\mathrm{Cl} 4$ | $95.69(2)$ |  |  |

H atoms were located from difference Fourier maps. For (I), (II), (III) and (V), H atoms were then placed in idealized positions [ $\mathrm{C}-$ $\mathrm{H}=0.95 \AA, U_{\text {iso }}(\mathrm{H})=1.25 U_{\text {eq }}(\mathrm{C})$ for CH and $\mathrm{CH}_{2}$, and $U_{\text {iso }}(\mathrm{H})=$ $1.5 U_{\text {eq }}(\mathrm{C})$ for $\left.\mathrm{CH}_{3}\right]$. For (IV), H atoms were refined isotropically, resulting in refined $\mathrm{C}-\mathrm{H}$ distances in the range 0.79 (2)-1.06 (2) $\AA$.

For all compounds, data collection: SMART (Siemens, 1995); cell refinement: SAINT (Siemens, 1995); data reduction: Xtal3.5 (Hall et al., 1995); structure solution: SIMPEL in Xtal3.5; structure refinement: CRYLSQ in Xtal3.5; molecular graphics: Xtal3.5; software used to prepare material for publication: BONDLA and CIFIO in Xtal3.5.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1697). Services for accessing these data are described at the back of the journal.

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