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Redetermination of silicon(IV) acetate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.001 Å
R factor = 0.023
wR factor = 0.063
Data-to-parameter ratio = 27.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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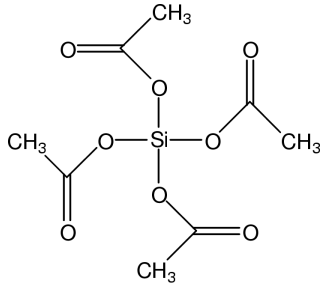
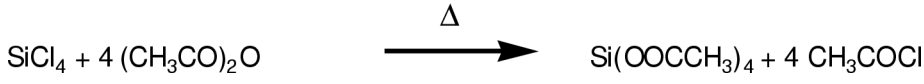
The title compound, $C_8H_{12}O_8Si$, previously reported by Kamenar & Bruvo [*Z. Kristallogr.* (1975). **141**, 97–103], has been rerefined against new intensity data. Geometric parameters agree quite well. However, our results are of significantly higher precision. The Si atom is located on a special position of site symmetry $\overline{4}$.

Comment

The original structure of the title compound, (I), was reported by Kamenar & Bruvo (1975) using Weissenberg photographs for the determination of the reflection intensities. The synthesis of (I) was achieved by transformation of $SiCl_4$ as indicated in the reaction Scheme below. The Si atom is located on a special position of site symmetry $\overline{4}$. All other atoms are located on general positions. There is only a quarter of a molecule in the asymmetric unit. The geometric parameters of both determinations agree quite well, but the present work is of significantly improved precision. In addition, we have determined the position of the H atoms. A perspective view of the title compound is shown in Fig. 1.



(I)



Experimental

The title compound, (I), was obtained by adding 7 ml $SiCl_4$ to a solution of 25 ml $(CH_3CO)_2O$ and 13 ml Et_2O at ambient temperature. After heating under reflux for 48 h, colourless crystals of (I) were grown by storing this solution at 298 K for 3 d. The NMR spectra were recorded on a Bruker DPX 250 spectrometer. ^{1}H NMR ($CDCl_3$, internal TMS, p.p.m.): δ 2.034 (*s*, 4 Me). $^{13}C\{^1H\}$ NMR ($CDCl_3$, internal TMS, p.p.m.): δ 22.2 (4 Me), δ 167.8 (4 COO). ^{29}Si NMR ($CDCl_3$, external TMS, p.p.m.): δ −96.4 (*s*).

Crystal data

$C_8H_{12}O_8Si$
$M_r = 264.27$
Tetragonal, $P\bar{4}2_1c$
$a = 7.3086\ (6)$ Å
$c = 11.2295\ (12)$ Å
$V = 599.83\ (9)$ Å^3
$Z = 2$
$D_x = 1.463$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10715 reflections
$\theta = 3.8$–$32.6°$
$\mu = 0.22$ mm^{-1}
$T = 173\ (2)$ K
Block, colourless
0.48 × 0.24 × 0.22 mm

Data collection

Stoe IPDS II two-circle diffractometer
ω scans
Absorption correction: empirical (*MULABS*; Spek, 1990; Blessing, 1995)
$T_{min} = 0.659$, $T_{max} = 0.952$
7495 measured reflections
1091 independent reflections
1059 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 32.7°$
$h = -11 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.063$
$S = 1.10$
1091 reflections
40 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.0161P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.084 (16)
Absolute structure: Flack (1983), 441 Friedel pairs
Flack parameter = 0.07 (13)

Table 1
Selected geometric parameters (Å, °).

Si1—O1	1.6380 (6)	C1—O2	1.2002 (8)
O1—C1	1.3640 (10)		
O1^{i}—Si1—O1	100.04 (4)	C1—O1—Si1	124.90 (5)
O1—Si1—O1^{ii}	114.38 (2)		

Symmetry codes: (i) $2-x, 2-y, z$; (ii) $y, 2-x, -z$.

All H atoms were located by difference Fourier syntheses. They were refined with fixed individual displacement parameters [U_{iso}(H) = 1.5U_{eq}(C)], using a riding model with C—H$_{methyl}$ = 0.98 Å.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

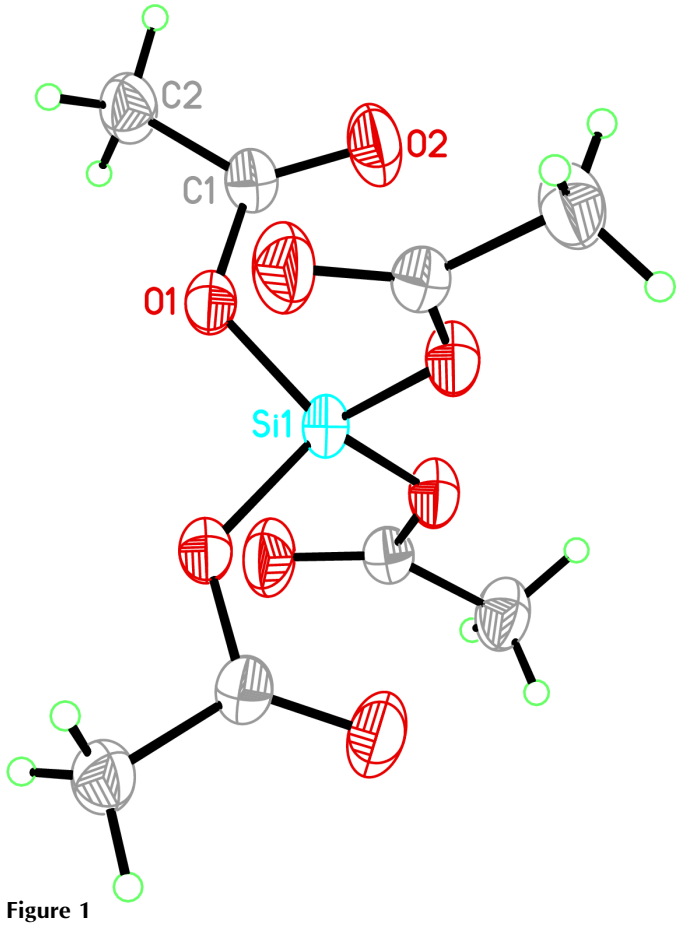


Figure 1
Perspective view of (I), with the atom numbering. Displacement ellipsoids are at the 50% probability level.

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