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Key indicators

Single-crystal X-ray study
 $T = 150\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.043
 wR factor = 0.122
Data-to-parameter ratio = 18.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

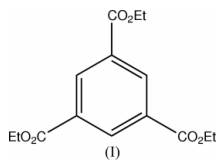
Triethyl benzene-1,3,5-tricarboxylate

The title compound, $\text{C}_{15}\text{H}_{18}\text{O}_6$, first synthesized in 1868, has previously been analysed by single-crystal X-ray diffraction, but only the unit-cell dimensions and space group have been published. Here we present the full low-temperature structure determination and show that the title compound crystallizes in the chiral space group $P6_5$ (or $P6_1$), despite its inherent non-chirality.

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Comment

The title compound, (I), was first synthesized in 1868 (Fittig & Furtenbach, 1868) and is the second simplest member of the known family of trimesic acid (benzene-1,3,5-tricarboxylic acid) alkyl triesters, which spans methyl (Karabinos *et al.*, 1946) through to tetracosyl (C_{24}) chains (Breusch & Ulusoy, 1961). None of these alkyl esters has been fully structurally characterized, although the more elaborate 1,3,5-tris[(4-tri-allylsilyl)phenylester]benzene (Cambridge Structural Database, Version 5.24, April 2003 update; Allen, 2002; refcode GUCQOK; Gossage *et al.*, 1999) has been analysed using the single-crystal X-ray technique. The trimethyl ester has been used as a ligand for such metals as Ti, Al and Re, for which the structures of the complexes are known (Sobota *et al.*, 1993; Utko *et al.*, 1993; Adams *et al.*, 1993).



The unit-cell dimensions and space group of (I) have been previously determined (Hertel & Dumont, 1935), but here the full low-temperature structure analysis is presented.

The initial solution, provided by *SIR92* (Altomare *et al.*, 1994), in space group $P3_2$ indicated two molecules in the asymmetric unit ($Z' = 2$); however, *PLATON* (Spek, 2003) identified additional missed symmetry, yielding the space group $P6_5$ (or $P6_1$; the absolute configuration was not determined) and hence $Z' = 1$.

The three independent carboxyl groups deviate little from coplanarity with the aromatic ring [dihedral angles with respect to ring C1–C6: C7–O1–O2 $3.76(3)^\circ$; C10–O3–O4 $4.38(4)^\circ$; C13–O5–O6 $0.56(2)^\circ$]; furthermore, the C–O and C=O bond lengths (Table 1) are in the expected ranges.

In the crystal structure, molecules of (I) form sheets in the *ab* plane.

Experimental

Catalytic H_2SO_4 was added to a refluxing ethanolic solution (25 ml) of trimesic acid (500 mg, 2.38 mmol). After 60 h, the solution was allowed to cool and the solvent removed *in vacuo*. The residue was dissolved in dichloromethane (10 ml) and washed with saturated aqueous NaHCO_3 (3×10 ml). The organic layer was then dried (Na_2SO_4), filtered and evaporated to dryness, yielding (I) (602 mg, 86%) as a white solid. X-ray quality crystals were grown by slow evaporation of a dichloromethane solution of (I). M.p. 405–407 K (literature 407 K; Ghosh *et al.*, 1999); analysis calculated for $\text{C}_{15}\text{H}_{18}\text{O}_6$: C 61.22, H 6.16%; found: C 61.08; H 6.18%. IR, ^1H NMR and ^{13}C NMR spectroscopy data agree with those previously reported (Ghosh *et al.*, 1999).

Crystal data

$\text{C}_{15}\text{H}_{18}\text{O}_6$	Mo $K\alpha$ radiation
$M_r = 294.29$	Cell parameters from 4977 reflections
Hexagonal, $P6_5$	$\theta = 2.3\text{--}27.9^\circ$
$a = 11.3438$ (17) Å	$\mu = 0.10$ mm $^{-1}$
$c = 19.665$ (4) Å	$T = 150$ (2) K
$V = 2191.5$ (6) Å 3	Block, colourless
$Z = 6$	$0.37 \times 0.25 \times 0.09$ mm
$D_x = 1.338$ Mg m $^{-3}$	

Data collection

Bruker SMART 1000 CCD diffractometer	3515 independent reflections
ω scans	2448 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$R_{\text{int}} = 0.038$
$T_{\text{min}} = 0.910$, $T_{\text{max}} = 0.991$	$\theta_{\text{max}} = 28.8^\circ$
19450 measured reflections	$h = -14 \rightarrow 14$
	$k = -14 \rightarrow 14$
	$l = -26 \rightarrow 26$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 1.115P]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.122$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.27$ e Å $^{-3}$
3515 reflections	$\Delta\rho_{\text{min}} = -0.24$ e Å $^{-3}$
194 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.0005 (2)

Table 1

Selected geometric parameters (Å).

C7—O1	1.211 (3)	C7—O2	1.338 (3)
C10—O3	1.208 (3)	C10—O4	1.339 (3)
C13—O5	1.212 (3)	C13—O6	1.337 (3)

All H atoms were placed in geometrically calculated positions, and were refined using a riding model (aromatic C—H distance = 0.95 Å; methylene and methyl C—H distances = 0.99 and 0.98 Å, respectively). U_{iso} values were set to $1.2U_{\text{eq}}$ for aryl H ($1.5U_{\text{eq}}$ for methyl H and methylene H). 1543 Friedel pairs were measured but these were not merged prior to refinement.

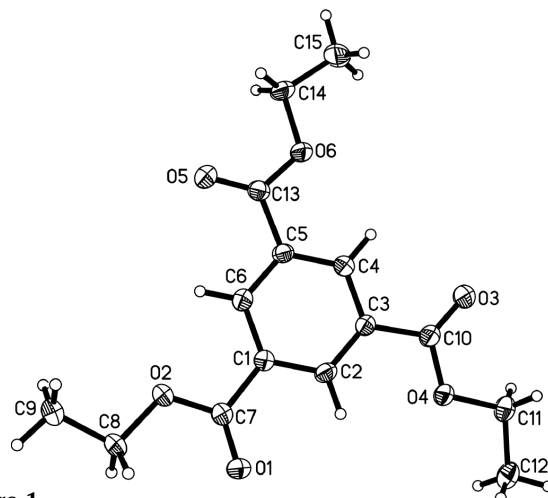


Figure 1

View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary radii.

Data collection: SMART (Siemens, 1994); cell refinement: SAINT (Siemens, 1994); data reduction: SAINT; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: SHELXTL (Bruker, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and local programs.

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References

- Adams, R. D., Chen, L. & Wu, W. (1993). *Organometallics*, **12**, 1623–1628.
- Allen, F. H. (2002). *Acta Cryst. B* **58**, 380–388.
- Altomare, A., Casciarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Breusch, F. L. & Ulusoy, E. (1961). *Istanbul Univ. Fen Fak. Mecmuası Seri C*, **26**, 1–5.
- Bruker (1997). SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.
- Fittig, R. & Furtenbach, E. v. (1868). *Justus Liebigs Ann. Chem.* **147**, 292–312.
- Ghosh, C. K., Bhattacharyya, S., Ghosh, C. & Patra, A. (1999). *J. Chem. Soc. Perkin Trans. 1*, pp. 3005–3013.
- Gossage, R. A., Muñoz-Martínez, E., Frey, H., Burgarth, A., Lutz, M., Spek, A. L. & von Kote, G. (1999). *Chem. Eur. J.* **5**, 2191–2197.
- Hertel, E. & Dumont, E. (1935). *Z. Phys. Chem. B* **29**, 112–116.
- Karabinos, J. V., Wright, J. I. & Hipsher, H. F. (1946). *J. Am. Chem. Soc.* **68**, 906.
- Sheldrick, G. M. (2001). SADABS. Version 2.03. University of Göttingen, Germany.
- Siemens (1994). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sobota, P., Utoko, J. & Lis, T. (1993). *J. Organomet. Chem.* **447**, 213–220.
- Spek, A. L. (2003). PLATON. Utrecht University, The Netherlands.
- Utoko, J., Wróblewska, M., Lis, T. & Sobota, P. (1993). *J. Organomet. Chem.* **447**, 159–161.