Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(2,4,6-trichlorophenyl) 2-ethylmalonate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.034
wR factor = 0.103
Data-to-parameter ratio = 13.7

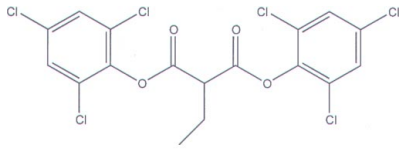
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 2 December 2003
Accepted 15 December 2003
Online 24 December 2003

2-Ethyl-1,3-propanedioic (malonic) acid forms a diester, bis(2,4,6-trichlorophenyl) ester, with 2,4,6-trichlorophenol to give the non-planar title compound, $C_{17}H_{10}Cl_6O_4$.

Comment

2,4,6-Trichlorophenyl esters of malonic acid, dubbed 'magic malonates', are particularly reactive (Kappe, 1967). Reactions of these esters are often carried out at 423–533 K in the condensed phase. It is possible that the high reactivity is due to aryloxycarbonyl ketene formation, especially in the higher-temperature range. The present paper reports the crystal structure determination for 2-ethylmalonic acid *syn*-bis(2,4,6-trichlorophenyl)ester, (I). The non-planar diester packs in the triclinic space group $P\overline{1}$ with a non-bonding distance of 3.437 (2) Å for Cl2 across the centre of symmetry (Table 1).



(I)

Experimental

The synthesis of (I) has been described by Kappe (1967).

Crystal data

$C_{17}H_{10}Cl_6O_4$
M_r = 490.95
Triclinic, $P\overline{1}$
a = 7.648 (3) Å
b = 8.532 (2) Å
c = 16.947 (6) Å
α = 91.41 (2)°
β = 92.98 (2)°
γ = 112.26 (2)°
V = 1020.8 (6) Å^3
Z = 2
D_x = 1.597 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.0–12.0°
μ = 0.86 mm^{-1}
T = 293 (2) K
Block, colourless
0.20 × 0.15 × 0.15 mm

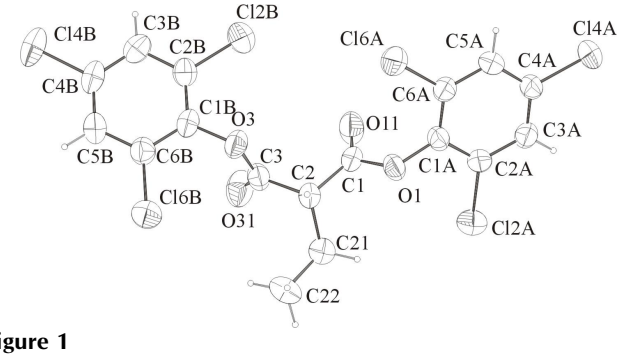


Figure 1
ORTEP plot (Spek, 1988) of (I), with displacement ellipsoids at the 50% probability level.

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.008
Non-profiled $\omega/2\theta$ scans	θ_{max} = 25.0°
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -1 \rightarrow 7$
T_{min} = 0.839, T_{max} = 0.879	$k = -10 \rightarrow 9$
3834 measured reflections	$l = -20 \rightarrow 20$
3352 independent reflections	3 standard reflections
2430 reflections with $I > 2\sigma(I)$	frequency: 60 min
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2 + 0.6749P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.103	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
S = 0.94	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
3352 reflections	
244 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.369 (3)	C3—O31	1.190 (3)
O1—C1*A*	1.384 (3)	C3—O3	1.345 (3)
C2—C3	1.516 (4)	O3—C1*B*	1.392 (3)
O11—C1—C2	129.1 (2)	O31—C3—C2	127.1 (3)
O1—C1—C2	108.4 (2)	O3—C3—C2	108.9 (2)
C1—O1—C1*A*	117.8 (2)	C3—O3—C1*B*	118.4 (2)
C1—C2—C3	108.8 (2)		
C2*A*—C1*A*—O1—C1	−101.4 (3)	C1—C2—C3—O3	−90.0 (3)
C1*A*—O1—C1—C2	177.8 (2)	C2—C3—O3—C1*B*	−178.6 (2)
O1—C1—C2—C3	163.6 (2)	C3—O3—C1*B*—C2*B*	−104.3 (3)

H atoms were treated as riding, with C—H distances in the range 0.93–0.98 Å and U_{iso}(H) values of $1.5U_{eq}$(C) for methyl H atoms and $1.2U_{eq}$(C) for all other H atoms.

Data collection: *SDP* (Frenz, 1985); cell refinement: *SDP*; data reduction: *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97; molecular graphics: *PLATON*98 (Spek, 2003); software used to prepare material for publication: *SHELXL*97.

The authors thank the Australian Research Council, the University of Queensland and Griffith University for financial support for the purchase of the CAD-4 diffractometer.

References

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Frenz, B. A. (1985). *SDP*. Enraf–Nonius, Delft, The Netherlands.
Kappe, T. (1967). *Monatsh. Chem.* **98**, 874–886.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1988). *J. Appl. Cryst.* **36**, 7–13.