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Tetraethyl pyrazine-2,3,5,6-tetracarboxylate

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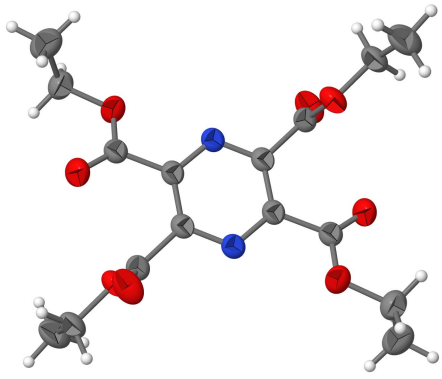
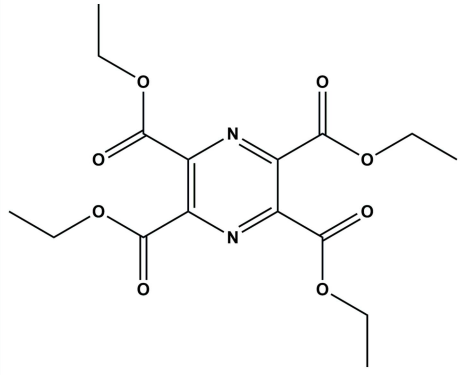
Structural data: full structural data are available from iucrdata.iucr.org

The whole molecule of the title compound, $C_{16}H_{20}N_2O_8$, is generated by inversion symmetry. The adjacent carboxylate groups [C(=O)—O—C] are inclined to the pyrazine ring by 72.40 (10) and 19.64 (10)°, and to one another by 68.21 (12)°. In the crystal, molecules stack along the *a*-axis direction but there are no significant intermolecular interactions present.

3D view



Chemical scheme



Synthesis and crystallization

The title compound (Fig. 1) was prepared by the method of Mager & Berends (1960). Colourless crystals were obtained by slow evaporation of a solution in THF (yield 71%; m.p. 375–377 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were included in calculated positions and treated as riding atoms: C—H = 0.96–0.97 Å with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms.

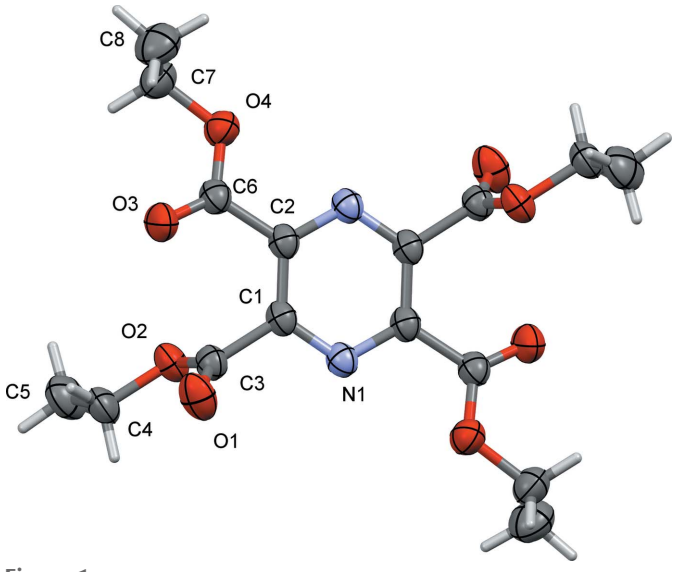


Figure 1
The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by the symmetry operation $-x, -y, -z + 1$.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{20}N_2O_8$
M_r	368.34
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	5.4194 (8), 9.0619 (12), 9.4607 (13)
α, β, γ (°)	81.285 (11), 82.771 (11), 77.208 (11)
V (Å^3)	445.80 (11)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.31 × 0.27 × 0.07
Data collection	
Diffractometer	STOE IPDS 2 diffractometer
Absorption correction	Multi-scan (*MULABS*; Spek, 2009)
T_{min}, T_{max}	0.650, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6815, 2408, 1414
R_{int}	0.078
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.687
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.124, 0.86
No. of reflections	2408
No. of parameters	121
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.21

Computer programs: *X-AREA* (Stoe & Cie, 2009), *X-RED32* (Stoe & Cie, 2009), *SHELXS2014/6* (Sheldrick, 2008), *SHELXL2014/6* (Sheldrick, 2015), *PLATON* (Spek, 2009), *Mercury* (Macrae *et al.*, 2008), *publCIF* (Westrip, 2010).

Acknowledgements

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full crystallographic data

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Crystal data

$C_{16}H_{20}N_2O_8$
M_r = 368.34
Triclinic, $P\overline{1}$
a = 5.4194 (8) Å
b = 9.0619 (12) Å
c = 9.4607 (13) Å
α = 81.285 (11)°
β = 82.771 (11)°
γ = 77.208 (11)°
V = 445.80 (11) Å^3
Z = 1
$F(000)$ = 194
D_x = 1.372 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4119 reflections
θ = 2.2–29.5°
μ = 0.11 mm^{-1}
T = 293 K
Plate, colourless
0.31 × 0.27 × 0.07 mm

Data collection

STOE IPDS 2 diffractometer
Radiation source: fine-focus sealed tube
Plane graphite monochromator
$\varphi + \omega$ scans
Absorption correction: multi-scan (MULABS; Spek, 2009)
T_{min} = 0.650, T_{max} = 1.000
6815 measured reflections
2408 independent reflections
1414 reflections with $I > 2\sigma(I)$
R_{int} = 0.078
θ_{max} = 29.2°, θ_{min} = 2.2°
h = −7→7
k = −12→12
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.124
S = 0.86
2408 reflections
121 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0654P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.054 (10)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.4462 (2)	−0.06169 (15)	0.77032 (15)	0.0584 (4)
O2	0.1176 (2)	0.13155 (13)	0.81276 (11)	0.0453 (3)
O3	0.4091 (2)	0.25124 (15)	0.54380 (14)	0.0565 (4)
O4	0.1520 (2)	0.36434 (13)	0.37225 (13)	0.0484 (3)
N1	0.0156 (2)	−0.10741 (14)	0.62316 (14)	0.0384 (3)
C1	0.1213 (3)	0.01462 (17)	0.60885 (16)	0.0361 (3)
C2	0.1086 (3)	0.12084 (17)	0.48617 (16)	0.0367 (3)
C3	0.2530 (3)	0.02424 (17)	0.73804 (17)	0.0396 (4)
C4	0.2194 (4)	0.1491 (2)	0.94406 (18)	0.0507 (4)
H4B	0.3871	0.1728	0.9215	0.061*
H4A	0.2333	0.0552	1.0100	0.061*
C5	0.0414 (5)	0.2756 (2)	1.0102 (2)	0.0679 (6)
H5A	−0.1260	0.2537	1.0266	0.102*
H5B	0.0379	0.3691	0.9466	0.102*
H5C	0.0975	0.2855	1.0999	0.102*
C6	0.2424 (3)	0.25202 (18)	0.47080 (17)	0.0406 (4)
C7	0.2689 (4)	0.4982 (2)	0.3480 (2)	0.0534 (4)
H7A	0.3113	0.5185	0.4388	0.064*
H7B	0.1485	0.5865	0.3092	0.064*
C8	0.5034 (4)	0.4738 (3)	0.2464 (2)	0.0704 (6)
H8A	0.4633	0.4482	0.1583	0.106*
H8B	0.6282	0.3920	0.2884	0.106*
H8C	0.5696	0.5655	0.2270	0.106*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0437 (7)	0.0671 (8)	0.0647 (8)	0.0053 (6)	−0.0225 (6)	−0.0197 (6)
O2	0.0438 (6)	0.0549 (6)	0.0382 (6)	−0.0040 (5)	−0.0117 (5)	−0.0120 (5)
O3	0.0576 (7)	0.0670 (8)	0.0542 (8)	−0.0311 (6)	−0.0183 (6)	0.0004 (6)
O4	0.0471 (6)	0.0465 (6)	0.0540 (7)	−0.0166 (5)	−0.0101 (5)	0.0001 (5)
N1	0.0335 (6)	0.0453 (7)	0.0377 (7)	−0.0098 (6)	−0.0046 (5)	−0.0062 (5)
C1	0.0289 (7)	0.0439 (8)	0.0364 (8)	−0.0067 (6)	−0.0028 (6)	−0.0092 (6)
C2	0.0304 (7)	0.0430 (8)	0.0381 (8)	−0.0088 (6)	−0.0026 (6)	−0.0089 (6)
C3	0.0359 (8)	0.0454 (8)	0.0398 (8)	−0.0111 (7)	−0.0059 (6)	−0.0066 (7)
C4	0.0603 (11)	0.0575 (10)	0.0386 (9)	−0.0127 (9)	−0.0153 (8)	−0.0101 (7)
C5	0.0869 (15)	0.0707 (12)	0.0470 (11)	−0.0080 (11)	−0.0073 (10)	−0.0220 (9)
C6	0.0380 (8)	0.0488 (9)	0.0373 (8)	−0.0135 (7)	−0.0018 (6)	−0.0078 (7)
C7	0.0575 (11)	0.0459 (9)	0.0594 (11)	−0.0184 (8)	−0.0047 (8)	−0.0042 (8)
C8	0.0699 (13)	0.0713 (13)	0.0711 (14)	−0.0281 (11)	0.0059 (11)	−0.0024 (11)

Geometric parameters (Å, º)

O1—C3	1.2012 (18)	C4—C5	1.487 (3)
O2—C3	1.3212 (19)	C4—H4B	0.9700

O2—C4	1.4642 (18)	C4—H4A	0.9700
O3—C6	1.2026 (19)	C5—H5A	0.9600
O4—C6	1.324 (2)	C5—H5B	0.9600
O4—C7	1.465 (2)	C5—H5C	0.9600
N1—C1	1.3359 (19)	C7—C8	1.490 (3)
N1—C2^{i}	1.3370 (19)	C7—H7A	0.9700
C1—C2	1.389 (2)	C7—H7B	0.9700
C1—C3	1.513 (2)	C8—H8A	0.9600
C2—N1^{i}	1.3370 (19)	C8—H8B	0.9600
C2—C6	1.504 (2)	C8—H8C	0.9600
C3—O2—C4	116.24 (12)	H5A—C5—H5B	109.5
C6—O4—C7	117.08 (13)	C4—C5—H5C	109.5
C1—N1—C2^{i}	116.40 (13)	H5A—C5—H5C	109.5
N1—C1—C2	122.26 (13)	H5B—C5—H5C	109.5
N1—C1—C3	113.45 (13)	O3—C6—O4	125.77 (15)
C2—C1—C3	124.29 (13)	O3—C6—C2	121.82 (15)
N1^{i}—C2—C1	121.33 (13)	O4—C6—C2	112.39 (13)
N1^{i}—C2—C6	118.01 (14)	O4—C7—C8	111.08 (15)
C1—C2—C6	120.63 (13)	O4—C7—H7A	109.4
O1—C3—O2	125.92 (15)	C8—C7—H7A	109.4
O1—C3—C1	123.30 (14)	O4—C7—H7B	109.4
O2—C3—C1	110.63 (12)	C8—C7—H7B	109.4
O2—C4—C5	107.54 (15)	H7A—C7—H7B	108.0
O2—C4—H4B	110.2	C7—C8—H8A	109.5
C5—C4—H4B	110.2	C7—C8—H8B	109.5
O2—C4—H4A	110.2	H8A—C8—H8B	109.5
C5—C4—H4A	110.2	C7—C8—H8C	109.5
H4B—C4—H4A	108.5	H8A—C8—H8C	109.5
C4—C5—H5A	109.5	H8B—C8—H8C	109.5
C4—C5—H5B	109.5		
C2^{i}—N1—C1—C2	−1.3 (2)	N1—C1—C3—O2	−105.99 (15)
C2^{i}—N1—C1—C3	178.62 (12)	C2—C1—C3—O2	73.95 (18)
N1—C1—C2—N1^{i}	1.4 (2)	C3—O2—C4—C5	179.40 (16)
C3—C1—C2—N1^{i}	−178.55 (13)	C7—O4—C6—O3	1.3 (2)
N1—C1—C2—C6	−176.59 (14)	C7—O4—C6—C2	−179.88 (13)
C3—C1—C2—C6	3.5 (2)	N1^{i}—C2—C6—O3	−160.17 (15)
C4—O2—C3—O1	2.1 (2)	C1—C2—C6—O3	17.9 (2)
C4—O2—C3—C1	177.82 (13)	N1^{i}—C2—C6—O4	21.0 (2)
N1—C1—C3—O1	69.8 (2)	C1—C2—C6—O4	−160.99 (14)
C2—C1—C3—O1	−110.23 (19)	C6—O4—C7—C8	84.0 (2)

Symmetry code: (i) $-x, -y, -z+1$.