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Key indicators

Single-crystal X-ray study
T = 294 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.041
wR factor = 0.107
Data-to-parameter ratio = 15.0

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

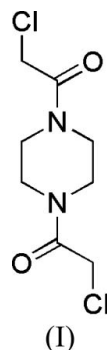
1,4-Bis(chloroacetyl)piperazine

The title compound, $\text{C}_8\text{H}_{12}\text{Cl}_2\text{N}_2\text{O}_2$, contains two half-molecules in the asymmetric unit. The complete molecule is generated by inversion symmetry in both cases.

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Comment

Piperazine derivatives have important applications as drugs and as synthetic intermediates (Halazy *et al.*, 1996; Krakowiak *et al.*, 1991). As part of our studies in this area, the title compound, (I), was synthesized from piperazine and chloroacetyl chloride. There are two half-molecules in the asymmetric unit. The complete molecule is generated by inversion symmetry in both cases, resulting in a typical chair conformation for the piperazine rings (Fig. 1). The conformations of the side chains of the two molecules are very similar (Table 1).



Experimental

Chloroacetyl chloride (54.2 g, 0.48 mol) was added dropwise to a solution of piperazine (17.2 g, 0.2 mol) in water (200 ml) at 263 K for 1 h. Na_2CO_3 (25.4 g, 0.24 mol) was then added. The reaction mixture was stirred at 263–268 K for a further 1 h. The precipitate was filtered

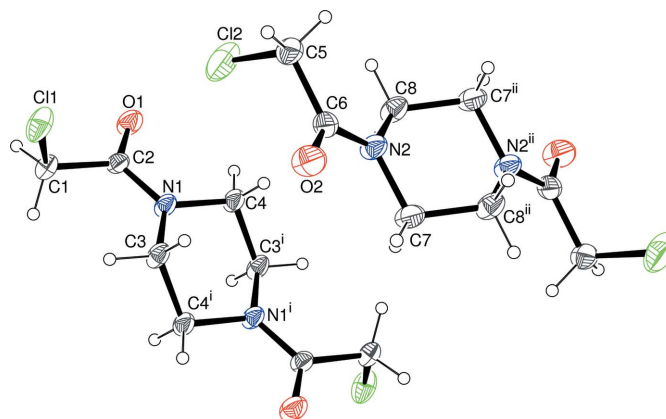


Figure 1

A view of the two independent molecules of (I), showing 30% probability displacement ellipsoids (arbitrary spheres for the H atoms). [Symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $1 - x, 1 - y, -z$.]

off, washed with water and dried *in vacuo*. Suitable crystals (yield 40.1 g, 83.9%; m.p. 408–410 K) of (I) were obtained by slow evaporation of solution in a mixture of dichloromethane and acetone (1:1 v/v).

Crystal data

$C_8H_{12}Cl_2N_2O_2$	$V = 543.2 (9) \text{ \AA}^3$
$M_r = 239.10$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.462 \text{ Mg m}^{-3}$
$a = 6.585 (6) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.339 (9) \text{ \AA}$	$\mu = 0.57 \text{ mm}^{-1}$
$c = 9.567 (9) \text{ \AA}$	$T = 294 (2) \text{ K}$
$\alpha = 69.501 (16)^\circ$	Block, colourless
$\beta = 89.001 (16)^\circ$	$0.26 \times 0.20 \times 0.18 \text{ mm}$
$\gamma = 80.591 (15)^\circ$	

Data collection

Bruker SMART CCD diffractometer	2747 measured reflections
ω scans	1908 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1469 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.865$, $T_{\max} = 0.904$	$R_{\text{int}} = 0.020$
	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.2514P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.107$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
1908 reflections	$\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$
127 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (\AA , $^\circ$).

C2–N1	1.359 (3)	C6–N2	1.360 (3)
C11–C1–C2–O1	–102.2 (2)	C12–C5–C6–O2	–102.5 (3)
C11–C1–C2–N1	78.4 (2)	C12–C5–C6–N2	77.7 (3)

All H atoms were positioned geometrically ($C-H = 0.97 \text{ \AA}$) and refined as riding, with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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