Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[4-(2-methyl-2-propenyl)piperazin-1-yl]-methane

Stefan Ricken, Markus Schürmann, Hans Preut* and Peter Eilbracht

Fachbereich Chemie, Universität Dortmund, Otto-Hahn-Strasse 6, 44221 Dortmund, Germany

Correspondence e-mail: uch002@uxp1.hrz.uni-dortmund.de

Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.083
Data-to-parameter ratio = 17.4

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

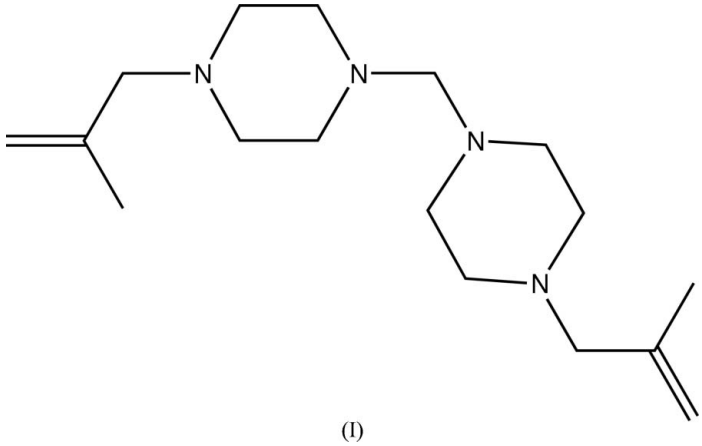
© 2006 International Union of Crystallography
All rights reserved

Received 3 March 2006
Accepted 8 March 2006

The title compound, $C_{17}H_{32}N_4$, contains two piperazine heterocycles, which are methylene bridged. The rings have chair conformations, with all substituents equatorial.

Comment

The title compound, (I), was formed from 1-(2-methyl-2-propenyl)piperazine, which was easily prepared from the mono-Boc-protected piperazine (Boschi *et al.*, 1994) and allylation of the second amine function with methallyl chloride and deprotection. 1-(2-Methyl-2-propenyl)piperazine is known and commercially available (CAS registry number: 57184–27-7) and was first synthesized by the Schering Corporation (Bloomfield & Sherlock, 1956) and later by Gao *et al.* (2003) by other methods.



(I)

We obtained (I) as colourless crystals after a flask was left to stand for 2–3 months at room temperature. The product could have been formed by the following process: after Boc removal with HCl in dioxane, the expected product was extracted with dichloromethane and the solvent was removed under reduced pressure. The ^{1}H NMR spectrum shows small amounts of dichloromethane. It is known that dichloromethane undergoes double nucleophilic substitution. The first reaction of this type was performed with dimethylamine (Jones & Whalen, 1925) and later with diethylamine (Souquet *et al.*, 1993; Matsumoto *et al.*, 1984). All these reactions were conducted at high temperature; in our case the double nucleophilic substitution occurs at room temperature with a reaction time of 2–3 months.

The crystal structure was determined primarily to identify the product. The two piperazine rings adopt the usual chair conformation, with all substituents equatorial. There are no significant intermolecular interactions.

Experimental

For the preparation of *tert*-butyl 4-(2-methylallyl)piperazine-1-carboxylate, Boc-piperazine (3 g, 16.1 mmol) was solved in toluene (30 ml) and triethylamine (2 ml). Next, 1.5 equivalents of methallyl chloride were added dropwise to the solution. The mixture was stirred under reflux for one day and the resulting solid was removed. The solvent was evaporated and the residue was purified by chromatography (diethyl ether) (yield 2.7 g, 70%). Spectroscopic data for all the prepared compounds are provided in the CIF.

For the preparation of 1-(2-methyl-2-propenyl)-piperazine, *tert*-butyl 4-(2-methylallyl)piperazine-1-carboxylate (3 g, 12.5 mmol) was dissolved in dioxane (30 ml) and 3 *M* HCl (30 ml) and the mixture stirred for 18 h. The solution was made basic with sodium hydroxide and the content of the flask was washed three times with dichloromethane (30 ml). The organic layer was dried over $MgSO_4$, filtered and the solvent removed under reduced pressure. 1-(2-Methyl-2-propenyl)piperazine (1.3 g) was formed as a colourless oil in 75% yield. Bis[4-(2-methyl-2-propenyl)piperazin-1-yl]methane, (I), was formed as long colourless needles after being left to stand for 2–3 months at room temperature in a flask.

Crystal data

$C_{17}H_{32}N_4$	$Z = 2$
$M_r = 292.47$	$D_x = 1.027$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 6.257$ (2) Å	Cell parameters from 6881 reflections
$b = 11.719$ (3) Å	$\theta = 3.1$–$25.2°$
$c = 13.392$ (5) Å	$\mu = 0.06$ mm^{-1}
$\alpha = 90.450$ (13)°	$T = 291$ (1) K
$\beta = 101.719$ (8)°	Block cut from needle, colourless
$\gamma = 100.067$ (15)°	0.20 × 0.15 × 0.15 mm
$V = 945.7$ (5) Å^3	

Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.033$
ω scans	$\theta_{max} = 25.2°$
Absorption correction: none	$h = -7 \rightarrow 7$
6681 measured reflections	$k = -13 \rightarrow 14$
3297 independent reflections	$l = -15 \rightarrow 15$
1123 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.038$	$w = [\exp\{4.2(\sin\theta/\lambda)^2\}]/[\sigma^2(F_o^2)]$
$wR(F^2) = 0.083$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.00$	$\Delta\rho_{max} = 0.09$ e Å^{-3}
3297 reflections	$\Delta\rho_{min} = -0.11$ e Å^{-3}
190 parameters	

H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and were refined as riding, with $U_{iso} = 1.5U_{eq}$ for methyl groups and $1.2U_{eq}$ for other H atoms; the methyl groups were allowed to rotate but not to tip.

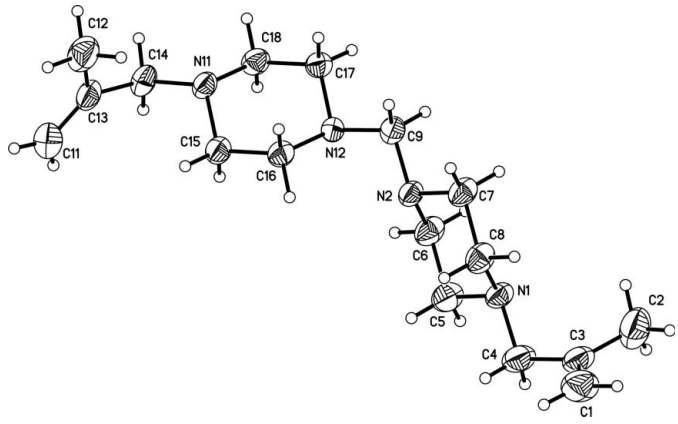


Figure 1
The molecular structure of the title compound, showing the labelling of all non-H atoms. Displacement ellipsoids are drawn at the 30% probability level.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*, *PARST95* (Nardelli, 1995) and *PLATON* (Spek, 2003).

References

Bloomfield, N. J. & Sherlock, M. H. (1956). US Pat. Appl. No. 2 899 431.
Boschi, D., Di Stilo, A., Fruttero, R., Medana, C., Sorba, G. & Gasco, A. (1994). *Arch. Pharm. (Weinheim)*, **327**, 661–667.
Gao, F. L., Wang, X., Zhang, H. M., Cheng, T. M. & Li, R. T. (2003). *Bioorg. Med. Chem. Lett.* **13**, 1535–1538.
Jones, L. W. & Whalen, H. F. (1925). *J. Am. Chem. Soc.* **47**, 1343–1352.
Matsumoto, K., Hashimoto, S., Ikemi, Y. & Otani, S. (1984). *Heterocycles*, **22**, 1417–1420.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1 Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Souquet, F., Martens, T. & Fleury, M.-B. (1993). *Synth. Commun.* **23**, 817–828.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.