

## *n*-Butyl 2-(2,4-dichloroanilino)-4,4-dimethyl-6-oxocyclohex-1-enecarbo-dithioate

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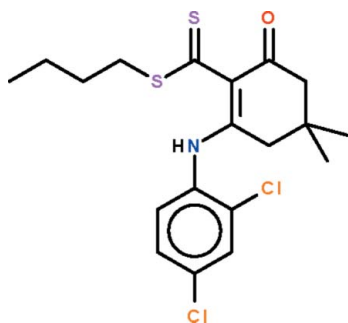
Received 8 September 2009; accepted 8 September 2009

Key indicators: single-crystal X-ray study;  $T = 123$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.025;  $wR$  factor = 0.070; data-to-parameter ratio = 19.8.

The cyclohexene ring in the title compound,  $\text{C}_{19}\text{H}_{23}\text{Cl}_2\text{NOS}_2$ , adopts an envelope conformation, with the C atom bearing the two methyl groups representing the flap. This atom deviates by 0.630 (2) Å from the plane passing through the other five atoms of the ring (r.m.s. deviation = 0.020 Å). The molecular conformation is stabilized by an intramolecular N—H...S hydrogen bond.

### Related literature

For the crystal structures of the *n*-undecanyl and 2-hydroxyethyl analogues, see: El Ashry *et al.* (2009*a,b*).



### Experimental

#### Crystal data

$\text{C}_{19}\text{H}_{23}\text{Cl}_2\text{NOS}_2$   
 $M_r = 416.40$   
Monoclinic,  $P2_1/c$   
 $a = 9.0321$  (1) Å  
 $b = 19.4422$  (2) Å  
 $c = 11.4700$  (1) Å  
 $\beta = 100.331$  (1)°

$V = 1981.52$  (3) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.55$  mm<sup>-1</sup>  
 $T = 123$  K  
0.30 × 0.20 × 0.10 mm

#### Data collection

Bruker SMART APEX  
diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.853$ ,  $T_{\max} = 0.947$

18747 measured reflections  
4554 independent reflections  
4279 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.016$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$   
 $wR(F^2) = 0.070$   
 $S = 1.00$   
4554 reflections  
230 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.41$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.25$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{S2}$	0.91 (2)	2.08 (2)	2.885 (1)	147 (2)

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINTE* (Bruker, 2008); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

The authors thank the University of Karachi and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2904).

### References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.  
Bruker (2008). *APEX2* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.  
El Ashry, E. S. H., Amer, M. R., Shah, M. R. & Ng, S. W. (2009*a*). *Acta Cryst.* **E65**, o601.  
El Ashry, E. S. H., Amer, M. R., Shah, M. R. & Ng, S. W. (2009*b*). *Acta Cryst.* **E65**, o602.  
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Westrip, S. P. (2009). *publCIF*. In preparation.

**supplementary materials**

*Acta Cryst.* (2009). E65, o2459 [ doi:10.1107/S1600536809036320 ]

***n*-Butyl 2-(2,4-dichloroanilino)-4,4-dimethyl-6-oxocyclohex-1-enecarbodithioate**

**E. S. H. El Ashry, M. R. Amer, M. Raza Shah and S. W. Ng**

**Experimental**

A cooled (283 K) solution of (2,4-dichloroanilino)-4,4-dimethyl-6-oxocyclohex-1-ene (0.2 mol) and sodium hydroxide (0.2 mol) in DMSO (30 ml) and water (2 ml), was treated with carbon disulfide (0.3 mol). After 40 min, *n*-bromobutane (0.15 mol) was added and the reaction mixture was left overnight. The mixture was then diluted with water (200 ml) and acidified with 10% hydrochloric acid. The product was purified on silica gel column chromatography to give yellow crystals when recrystallized from ethanol (m.p. 401 K).

**Refinement**

The N-bound H atom was located in a difference Fourier map and was refined freely. C-bound H atoms were placed in calculated positions (C—H = 0.95–0.99 Å) and were included in the refinement in the riding model approximation, with  $U_{iso}(H)$  set to 1.2 to 1.5 $U_{eq}(C)$ .

**Figures**

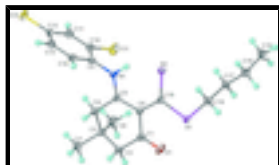


Fig. 1. Displacement ellipsoid plot (Barbour, 2001) plot of C<sub>19</sub>H<sub>23</sub>Cl<sub>2</sub>NOS<sub>2</sub> at the 70% probability level; H atoms are drawn as spheres of arbitrary radius.

***n*-Butyl 2-(2,4-dichloroanilino)-4,4-dimethyl-6-oxocyclohex-1-enecarbodithioate**

*Crystal data*

C<sub>19</sub>H<sub>23</sub>Cl<sub>2</sub>NOS<sub>2</sub>

$M_r = 416.40$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.0321 (1) \text{ \AA}$

$b = 19.4422 (2) \text{ \AA}$

$c = 11.4700 (1) \text{ \AA}$

$\beta = 100.331 (1)^\circ$

$V = 1981.52 (3) \text{ \AA}^3$

$Z = 4$

$F_{000} = 872$

$D_x = 1.396 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 9957 reflections

$\theta = 2.5\text{--}28.2^\circ$

$\mu = 0.55 \text{ mm}^{-1}$

$T = 123 \text{ K}$

Block, yellow

$0.30 \times 0.20 \times 0.10 \text{ mm}$

## Data collection

Bruker SMART APEX diffractometer	4554 independent reflections
Radiation source: fine-focus sealed tube	4279 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.016$
$T = 123$ K	$\theta_{\text{max}} = 27.5^\circ$
$\omega$ scans	$\theta_{\text{min}} = 2.1^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -11 \rightarrow 11$
$T_{\text{min}} = 0.853$ , $T_{\text{max}} = 0.947$	$k = -25 \rightarrow 25$
18747 measured reflections	$l = -14 \rightarrow 14$

## Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.025$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.070$	$w = 1/[\sigma^2(F_o^2) + (0.0405P)^2 + 0.9836P]$
$S = 1.00$	where $P = (F_o^2 + 2F_c^2)/3$
4554 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
230 parameters	$\Delta\rho_{\text{max}} = 0.41 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.25 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

## Special details

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

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.66246 (3)	0.420713 (17)	0.99199 (3)	0.02548 (8)
Cl2	1.25630 (3)	0.391875 (17)	1.00782 (3)	0.02572 (8)
S1	0.17044 (3)	0.547076 (14)	0.56447 (2)	0.01521 (7)
S2	0.45310 (3)	0.595199 (14)	0.71292 (3)	0.01818 (8)
O1	0.18119 (12)	0.41970 (5)	0.52118 (12)	0.0435 (3)
N1	0.65717 (11)	0.47990 (5)	0.74995 (8)	0.01551 (19)
H1	0.625 (2)	0.5237 (10)	0.7584 (16)	0.033 (4)*
C1	0.31311 (14)	0.40793 (6)	0.56335 (11)	0.0194 (2)
C2	0.37252 (13)	0.33742 (6)	0.54278 (10)	0.0176 (2)

H2A	0.4118	0.3381	0.4676	0.021*
H2B	0.2878	0.3044	0.5331	0.021*
C3	0.49613 (12)	0.31158 (6)	0.64090 (10)	0.0150 (2)
C4	0.61567 (12)	0.36787 (6)	0.66192 (10)	0.0163 (2)
H4A	0.6925	0.3547	0.7312	0.020*
H4B	0.6662	0.3698	0.5922	0.020*
C5	0.55899 (12)	0.43880 (6)	0.68327 (9)	0.0133 (2)
C6	0.41136 (12)	0.46044 (6)	0.63000 (10)	0.0141 (2)
C7	0.56586 (14)	0.24599 (6)	0.60044 (11)	0.0203 (2)
H7A	0.6051	0.2554	0.5277	0.030*
H7B	0.4890	0.2100	0.5851	0.030*
H7C	0.6482	0.2306	0.6626	0.030*
C8	0.43521 (15)	0.29707 (7)	0.75460 (11)	0.0246 (3)
H8A	0.3590	0.2607	0.7397	0.037*
H8B	0.3898	0.3390	0.7800	0.037*
H8C	0.5178	0.2822	0.8170	0.037*
C9	0.80208 (12)	0.45912 (6)	0.81146 (10)	0.0144 (2)
C10	0.81793 (13)	0.43077 (6)	0.92496 (10)	0.0160 (2)
C11	0.95775 (13)	0.40992 (6)	0.98561 (10)	0.0171 (2)
H11	0.9681	0.3902	1.0624	0.021*
C12	1.08177 (13)	0.41848 (6)	0.93158 (10)	0.0171 (2)
C13	1.07029 (13)	0.44805 (6)	0.82040 (10)	0.0187 (2)
H13	1.1572	0.4543	0.7856	0.022*
C14	0.92928 (13)	0.46846 (6)	0.76063 (10)	0.0169 (2)
H14	0.9198	0.4889	0.6845	0.020*
C15	0.35496 (12)	0.53004 (6)	0.63903 (9)	0.0140 (2)
C16	0.14330 (13)	0.63819 (6)	0.58501 (10)	0.0162 (2)
H16A	0.2411	0.6618	0.5885	0.019*
H16B	0.0737	0.6561	0.5150	0.019*
C17	0.08095 (13)	0.65650 (6)	0.69585 (10)	0.0169 (2)
H17A	0.1575	0.6462	0.7669	0.020*
H17B	-0.0089	0.6280	0.6994	0.020*
C18	0.03800 (14)	0.73269 (6)	0.69634 (11)	0.0226 (2)
H18A	0.1254	0.7609	0.6846	0.027*
H18B	-0.0451	0.7418	0.6292	0.027*
C19	-0.01100 (17)	0.75431 (7)	0.81137 (13)	0.0308 (3)
H19A	-0.0374	0.8033	0.8074	0.046*
H19B	0.0716	0.7464	0.8780	0.046*
H19C	-0.0988	0.7272	0.8227	0.046*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cl1	0.01664 (14)	0.03539 (18)	0.02576 (15)	-0.00287 (11)	0.00749 (11)	0.00779 (12)
Cl2	0.01535 (14)	0.03229 (17)	0.02687 (16)	0.00715 (11)	-0.00338 (11)	0.00089 (12)
S1	0.01301 (13)	0.01582 (14)	0.01536 (13)	0.00136 (9)	-0.00128 (10)	-0.00127 (9)
S2	0.01510 (14)	0.01493 (14)	0.02250 (15)	-0.00004 (10)	-0.00210 (11)	-0.00381 (10)
O1	0.0189 (5)	0.0256 (5)	0.0756 (8)	0.0054 (4)	-0.0192 (5)	-0.0213 (5)

## supplementary materials

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N1	0.0130 (4)	0.0142 (5)	0.0177 (5)	-0.0003 (3)	-0.0015 (3)	-0.0014 (4)
C1	0.0162 (5)	0.0169 (5)	0.0229 (6)	0.0000 (4)	-0.0023 (4)	-0.0034 (4)
C2	0.0170 (5)	0.0148 (5)	0.0188 (5)	-0.0003 (4)	-0.0028 (4)	-0.0027 (4)
C3	0.0135 (5)	0.0141 (5)	0.0162 (5)	-0.0014 (4)	0.0000 (4)	0.0001 (4)
C4	0.0123 (5)	0.0148 (5)	0.0210 (5)	-0.0003 (4)	0.0012 (4)	-0.0022 (4)
C5	0.0128 (5)	0.0150 (5)	0.0125 (5)	-0.0017 (4)	0.0028 (4)	0.0006 (4)
C6	0.0122 (5)	0.0145 (5)	0.0150 (5)	-0.0011 (4)	0.0008 (4)	-0.0008 (4)
C7	0.0188 (5)	0.0145 (5)	0.0259 (6)	-0.0003 (4)	-0.0010 (4)	-0.0021 (4)
C8	0.0264 (6)	0.0257 (6)	0.0225 (6)	-0.0027 (5)	0.0066 (5)	0.0050 (5)
C9	0.0124 (5)	0.0139 (5)	0.0156 (5)	-0.0009 (4)	-0.0009 (4)	-0.0023 (4)
C10	0.0138 (5)	0.0169 (5)	0.0175 (5)	-0.0024 (4)	0.0035 (4)	-0.0003 (4)
C11	0.0181 (6)	0.0169 (5)	0.0153 (5)	-0.0003 (4)	0.0002 (4)	0.0009 (4)
C12	0.0128 (5)	0.0178 (5)	0.0186 (5)	0.0025 (4)	-0.0028 (4)	-0.0027 (4)
C13	0.0142 (5)	0.0239 (6)	0.0183 (5)	-0.0002 (4)	0.0037 (4)	-0.0027 (4)
C14	0.0169 (5)	0.0197 (5)	0.0137 (5)	-0.0016 (4)	0.0010 (4)	-0.0012 (4)
C15	0.0126 (5)	0.0166 (5)	0.0125 (5)	-0.0006 (4)	0.0013 (4)	0.0003 (4)
C16	0.0176 (5)	0.0144 (5)	0.0158 (5)	0.0018 (4)	0.0007 (4)	0.0019 (4)
C17	0.0156 (5)	0.0163 (5)	0.0184 (5)	0.0011 (4)	0.0020 (4)	-0.0009 (4)
C18	0.0224 (6)	0.0180 (6)	0.0251 (6)	0.0033 (4)	-0.0016 (5)	-0.0030 (4)
C19	0.0309 (7)	0.0264 (7)	0.0340 (7)	0.0087 (5)	0.0030 (6)	-0.0091 (6)

### *Geometric parameters (Å, °)*

C11—C10	1.7287 (11)	C8—H8A	0.98
C12—C12	1.7387 (12)	C8—H8B	0.98
S1—C15	1.7628 (11)	C8—H8C	0.98
S1—C16	1.8094 (12)	C9—C14	1.3903 (16)
S2—C15	1.6851 (11)	C9—C10	1.3973 (16)
O1—C1	1.2244 (16)	C10—C11	1.3885 (16)
N1—C5	1.3291 (14)	C11—C12	1.3840 (17)
N1—C9	1.4293 (14)	C11—H11	0.95
N1—H1	0.911 (18)	C12—C13	1.3856 (17)
C1—C6	1.4733 (15)	C13—C14	1.3916 (16)
C1—C2	1.5061 (16)	C13—H13	0.95
C2—C3	1.5214 (15)	C14—H14	0.95
C2—H2A	0.99	C16—C17	1.5222 (16)
C2—H2B	0.99	C16—H16A	0.99
C3—C4	1.5258 (15)	C16—H16B	0.99
C3—C8	1.5300 (16)	C17—C18	1.5315 (16)
C3—C7	1.5307 (16)	C17—H17A	0.99
C4—C5	1.5063 (15)	C17—H17B	0.99
C4—H4A	0.99	C18—C19	1.5240 (19)
C4—H4B	0.99	C18—H18A	0.99
C5—C6	1.4269 (15)	C18—H18B	0.99
C6—C15	1.4562 (15)	C19—H19A	0.98
C7—H7A	0.98	C19—H19B	0.98
C7—H7B	0.98	C19—H19C	0.98
C7—H7C	0.98		
C15—S1—C16	105.02 (5)	C14—C9—N1	120.57 (10)

C5—N1—C9	124.86 (10)	C10—C9—N1	120.21 (10)
C5—N1—H1	115.5 (11)	C11—C10—C9	120.93 (10)
C9—N1—H1	119.6 (11)	C11—C10—C11	118.85 (9)
O1—C1—C6	121.90 (11)	C9—C10—C11	120.22 (9)
O1—C1—C2	117.19 (11)	C12—C11—C10	118.49 (10)
C6—C1—C2	120.90 (10)	C12—C11—H11	120.8
C1—C2—C3	114.80 (9)	C10—C11—H11	120.8
C1—C2—H2A	108.6	C11—C12—C13	121.94 (11)
C3—C2—H2A	108.6	C11—C12—C12	118.20 (9)
C1—C2—H2B	108.6	C13—C12—C12	119.85 (9)
C3—C2—H2B	108.6	C12—C13—C14	118.85 (11)
H2A—C2—H2B	107.5	C12—C13—H13	120.6
C2—C3—C4	106.54 (9)	C14—C13—H13	120.6
C2—C3—C8	111.29 (10)	C9—C14—C13	120.55 (11)
C4—C3—C8	110.49 (10)	C9—C14—H14	119.7
C2—C3—C7	109.76 (9)	C13—C14—H14	119.7
C4—C3—C7	109.11 (9)	C6—C15—S2	125.12 (8)
C8—C3—C7	109.58 (10)	C6—C15—S1	116.90 (8)
C5—C4—C3	115.52 (9)	S2—C15—S1	117.97 (6)
C5—C4—H4A	108.4	C17—C16—S1	114.63 (8)
C3—C4—H4A	108.4	C17—C16—H16A	108.6
C5—C4—H4B	108.4	S1—C16—H16A	108.6
C3—C4—H4B	108.4	C17—C16—H16B	108.6
H4A—C4—H4B	107.5	S1—C16—H16B	108.6
N1—C5—C6	123.03 (10)	H16A—C16—H16B	107.6
N1—C5—C4	115.65 (10)	C16—C17—C18	111.18 (10)
C6—C5—C4	121.26 (10)	C16—C17—H17A	109.4
C5—C6—C15	123.74 (10)	C18—C17—H17A	109.4
C5—C6—C1	116.59 (10)	C16—C17—H17B	109.4
C15—C6—C1	119.67 (10)	C18—C17—H17B	109.4
C3—C7—H7A	109.5	H17A—C17—H17B	108.0
C3—C7—H7B	109.5	C19—C18—C17	112.52 (11)
H7A—C7—H7B	109.5	C19—C18—H18A	109.1
C3—C7—H7C	109.5	C17—C18—H18A	109.1
H7A—C7—H7C	109.5	C19—C18—H18B	109.1
H7B—C7—H7C	109.5	C17—C18—H18B	109.1
C3—C8—H8A	109.5	H18A—C18—H18B	107.8
C3—C8—H8B	109.5	C18—C19—H19A	109.5
H8A—C8—H8B	109.5	C18—C19—H19B	109.5
C3—C8—H8C	109.5	H19A—C19—H19B	109.5
H8A—C8—H8C	109.5	C18—C19—H19C	109.5
H8B—C8—H8C	109.5	H19A—C19—H19C	109.5
C14—C9—C10	119.20 (10)	H19B—C19—H19C	109.5
O1—C1—C2—C3	-149.43 (13)	C14—C9—C10—C11	-2.07 (17)
C6—C1—C2—C3	31.47 (16)	N1—C9—C10—C11	179.58 (10)
C1—C2—C3—C4	-52.05 (13)	C14—C9—C10—C11	178.25 (9)
C1—C2—C3—C8	68.46 (13)	N1—C9—C10—C11	-0.10 (15)
C1—C2—C3—C7	-170.06 (10)	C9—C10—C11—C12	0.65 (17)
C2—C3—C4—C5	52.18 (12)	C11—C10—C11—C12	-179.66 (9)

## supplementary materials

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C8—C3—C4—C5	-68.83 (13)	C10—C11—C12—C13	1.04 (17)
C7—C3—C4—C5	170.62 (9)	C10—C11—C12—C12	-179.71 (9)
C9—N1—C5—C6	175.52 (10)	C11—C12—C13—C14	-1.26 (18)
C9—N1—C5—C4	-7.29 (16)	C12—C12—C13—C14	179.50 (9)
C3—C4—C5—N1	151.63 (10)	C10—C9—C14—C13	1.84 (17)
C3—C4—C5—C6	-31.13 (15)	N1—C9—C14—C13	-179.82 (10)
N1—C5—C6—C15	2.50 (17)	C12—C13—C14—C9	-0.21 (17)
C4—C5—C6—C15	-174.53 (10)	C5—C6—C15—S2	-0.17 (16)
N1—C5—C6—C1	-177.12 (10)	C1—C6—C15—S2	179.44 (9)
C4—C5—C6—C1	5.84 (15)	C5—C6—C15—S1	178.79 (8)
O1—C1—C6—C5	174.85 (13)	C1—C6—C15—S1	-1.60 (14)
C2—C1—C6—C5	-6.09 (16)	C16—S1—C15—C6	-175.86 (8)
O1—C1—C6—C15	-4.79 (19)	C16—S1—C15—S2	3.18 (8)
C2—C1—C6—C15	174.27 (10)	C15—S1—C16—C17	-89.34 (9)
C5—N1—C9—C14	95.10 (14)	S1—C16—C17—C18	-170.96 (8)
C5—N1—C9—C10	-86.58 (14)	C16—C17—C18—C19	-174.62 (10)

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1 $\cdots$ S2	0.91 (2)	2.08 (2)	2.885 (1)	147 (2)



Fig. 1

