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N-(Biphenyl-4-carbonyl)-*N*′-(4-chlorophenyl)thiourea

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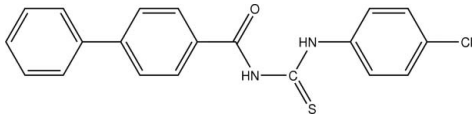
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.049; wR factor = 0.122; data-to-parameter ratio = 15.4.

In the title compound, $C_{20}H_{15}ClN_2OS$, the benzene rings of the biphenyl group are at an angle of 44.23 (12)°. The C_4N_2OS central thiourea fragment makes dihedral angles with the benzene carbonyl and chlorobenzene rings of 55.96 (9) and 64.09 (9)°, respectively. The *trans–cis* geometry of the thiourea group is stabilized by the intramolecular hydrogen bond between the carbonyl O atom and the H atom of the *cis*-thioamide. In the crystal structure, molecules are linked by N—H···S and N—H···O intermolecular hydrogen bonds to form one-dimensional chains along the *c* axis. C—H···π interactions also contribute to the stability of the molecule.

Related literature

For related literature, see: Allen *et al.* (1987); Arif & Yamin (2007).



Experimental

Crystal data

$C_{20}H_{15}ClN_2OS$
M_r = 366.85
Monoclinic, $P2/c$
a = 16.039 (7) Å
b = 6.087 (3) Å
c = 18.096 (8) Å
β = 94.780 (8)°
V = 1760.5 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 298 (2) K
0.49 × 0.46 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.848, T_{max} = 0.966
9371 measured reflections
3475 independent reflections
2278 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.122
S = 1.02
3475 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	2.00	2.683 (3)	135
N1—H1···S1^{i}	0.86	2.55	3.362 (3)	157
N2—H2···O1^{ii}	0.86	2.58	3.210 (3)	131
C1—H1*A*···*Cg*3^{ii}	0.93	2.98	3.586 (3)	124

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+2, y, -z+\frac{1}{2}$. *Cg*3 is the centroid of atoms C15–C20.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2027).

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supplementary materials

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N-(Biphenyl-4-carbonyl)-*N*'-(4-chlorophenyl)thiourea

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Comment

The title compound (Fig. 1) is an isomeric analog of the previously reported *N*-(biphenyl-4-carbonyl)-*N*'-(2-chlorophenyl) thiourea (II) (Arif and Yamin, 2007). The dihedral angle between the two benzene rings in the biphenyl fragment is 44.23 (12)°, which is double the value of 20.71 (17)° in (II). The examination on the planarity of the central thiourea fragment S1/N1/N2/C14 and the chlorophenyl plane (C15—C20)/Cl1, indicates that they are planar. The central thiourea fragment makes dihedral angles with the benzene carbonyl and chlorobenzene rings of 55.96 (9) and 64.09 (9)°, respectively. The *trans-cis* geometry in the thiourea moiety is stabilized by the N2—H2···O1 intramolecular hydrogen bond (Table 1).

In the crystal structure symmetry related molecules are linked by N1—H1···S1^{i} and N2—H2···O1^{ii} intermolecular hydrogen bonds to form one-dimensional chains along the *c* axis (Fig. 2 and Table 1). The molecule is also stabilized by a C1—H1A···π interaction; the distance between H1A and the (C15—C20) ring centroid is 2.98 Å, and the angle about the hydrogen atom is 124°.

Experimental

A solution of 4-chloroaniline (0.63 g, 2.5 mmol) in 20 ml acetone was added dropwise to a two-necked round-bottomed flask containing an equimolar amount of biphenylcarbomoylisothiocyanate (0.60 g, 2.5 mmol) in 20 ml of acetone. The mixture was refluxed for about 3 h. The light yellow solution was filtered and the filtrate allowed to evaporate at room temperature. Colourless crystals were obtained after five days (yield 0.71 g, 85%, m.p.: 164–166°C).

Refinement

H atoms on C and N atoms were positioned geometrically with C—H = 0.93 and N—H = 0.86 Å, and constrained to ride on their parent atoms with U_{iso}(H)= 1.2U_{eq}(parent atom).

Figures

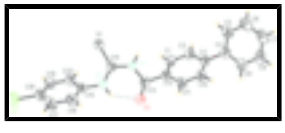

Fig. 1. Molecular structure of compound (I), with displacement ellipsoid drawn at the 50% probablity level. The dashed line indicates the intramolecular hydrogen bond.

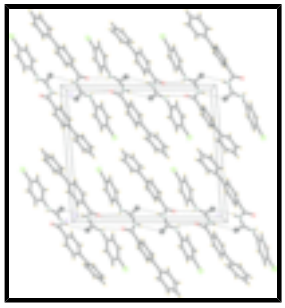

Fig. 2. A view along the *b* axis of the crystal packing of compound (I). The dashed line indicates the intermolecular N—H···S and N—H···O hydrogen bonds (see Table 1 for details).

N-(Biphenyl-4-carbonyl)-N'-(4-chlorophenyl)thiourea

Crystal data

$C_{20}H_{15}ClN_2OS$	F_{000} = 760
M_r = 366.85	D_x = 1.384 Mg m^{-3}
Monoclinic, *P*2/*c*	Melting point: 164-166°C K
Hall symbol: -P 2yc	Mo *K*α radiation λ = 0.71073 Å
a = 16.039 (7) Å	Cell parameters from 1713 reflections
b = 6.087 (3) Å	θ = 2.2–26.0º
c = 18.096 (8) Å	μ = 0.35 mm^{-1}
β = 94.780 (8)º	*T* = 298 (2) K
V = 1760.5 (14) Å^3	Block, colourless
Z = 4	0.49 × 0.46 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3475 independent reflections
Radiation source: fine-focus sealed tube	2278 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
T = 298(2) K	θ_{max} = 26.0º
ω scan	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	*h* = −19→18
T_{min} = 0.848, T_{max} = 0.966	*k* = −7→7
9371 measured reflections	*l* = −22→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.049	H-atom parameters constrained
$wR(F^2)$ = 0.122	$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.0946P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = <0.001

3475 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
226 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	1.37861 (5)	0.39472 (17)	0.18867 (5)	0.1065 (4)
S1	1.08379 (4)	0.38002 (11)	0.42312 (3)	0.0553 (2)
O1	0.95079 (10)	0.9893 (3)	0.32501 (9)	0.0591 (5)
N1	0.97515 (11)	0.7034 (3)	0.40513 (10)	0.0483 (5)
H1	0.9564	0.6440	0.4435	0.058*
N2	1.07779 (10)	0.7027 (3)	0.32382 (10)	0.0487 (5)
H2	1.0537	0.8194	0.3057	0.058*
C1	0.79626 (14)	0.7695 (4)	0.42417 (12)	0.0505 (6)
H1A	0.8026	0.6333	0.4020	0.061*
C2	0.72746 (13)	0.8084 (4)	0.46287 (13)	0.0504 (6)
H2A	0.6872	0.6993	0.4656	0.060*
C3	0.71742 (13)	1.0086 (4)	0.49788 (12)	0.0445 (6)
C4	0.64556 (14)	1.0439 (4)	0.54340 (13)	0.0481 (6)
C5	0.62285 (15)	0.8839 (4)	0.59148 (14)	0.0601 (7)
H5	0.6532	0.7538	0.5957	0.072*
C6	0.55587 (17)	0.9127 (5)	0.63367 (16)	0.0752 (9)
H6	0.5416	0.8032	0.6661	0.090*
C7	0.51023 (17)	1.1049 (6)	0.62738 (16)	0.0749 (9)
H7	0.4645	1.1246	0.6550	0.090*
C8	0.53267 (16)	1.2669 (5)	0.58009 (16)	0.0759 (9)
H8	0.5026	1.3976	0.5763	0.091*
C9	0.59943 (15)	1.2364 (5)	0.53843 (15)	0.0627 (7)
H9	0.6139	1.3468	0.5064	0.075*
C10	0.77729 (14)	1.1700 (4)	0.49070 (13)	0.0525 (6)
H10	0.7713	1.3058	0.5132	0.063*
C11	0.84582 (14)	1.1326 (4)	0.45067 (13)	0.0519 (6)
H11	0.8850	1.2432	0.4458	0.062*
C12	0.85579 (13)	0.9293 (4)	0.41789 (12)	0.0431 (5)

C13	0.93135 (13)	0.8825 (4)	0.37742 (12)	0.0451 (6)
C14	1.04533 (13)	0.6039 (4)	0.38029 (12)	0.0425 (5)
C15	1.15067 (13)	0.6247 (4)	0.29167 (11)	0.0426 (5)
C16	1.22033 (14)	0.7565 (5)	0.29525 (12)	0.0572 (7)
H16	1.2199	0.8927	0.3185	0.069*
C17	1.29127 (15)	0.6842 (5)	0.26385 (15)	0.0679 (8)
H17	1.3389	0.7719	0.2655	0.081*
C18	1.29059 (15)	0.4827 (5)	0.23047 (14)	0.0605 (7)
C19	1.22169 (16)	0.3514 (4)	0.22703 (14)	0.0594 (7)
H19	1.2226	0.2141	0.2046	0.071*
C20	1.15066 (15)	0.4242 (4)	0.25714 (13)	0.0530 (6)
H20	1.1027	0.3376	0.2541	0.064*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0737 (5)	0.1498 (9)	0.1022 (7)	0.0428 (5)	0.0449 (5)	0.0262 (6)
S1	0.0576 (4)	0.0604 (4)	0.0499 (4)	0.0152 (3)	0.0171 (3)	0.0127 (3)
O1	0.0684 (11)	0.0584 (11)	0.0528 (10)	0.0138 (9)	0.0183 (8)	0.0152 (9)
N1	0.0496 (11)	0.0554 (13)	0.0417 (11)	0.0119 (10)	0.0137 (9)	0.0092 (9)
N2	0.0495 (11)	0.0530 (12)	0.0454 (11)	0.0088 (9)	0.0138 (9)	0.0096 (10)
C1	0.0504 (14)	0.0455 (15)	0.0549 (15)	0.0053 (12)	0.0007 (11)	−0.0092 (12)
C2	0.0434 (13)	0.0483 (15)	0.0594 (15)	−0.0066 (11)	0.0039 (11)	−0.0050 (12)
C3	0.0415 (12)	0.0462 (15)	0.0456 (13)	0.0027 (11)	0.0029 (10)	0.0012 (11)
C4	0.0419 (13)	0.0507 (15)	0.0519 (14)	−0.0052 (11)	0.0046 (10)	−0.0057 (12)
C5	0.0554 (15)	0.0614 (18)	0.0644 (17)	−0.0039 (13)	0.0110 (13)	0.0014 (14)
C6	0.0655 (18)	0.090 (2)	0.0726 (19)	−0.0176 (17)	0.0198 (15)	0.0045 (17)
C7	0.0504 (16)	0.100 (3)	0.077 (2)	−0.0100 (17)	0.0222 (14)	−0.0116 (19)
C8	0.0542 (17)	0.084 (2)	0.091 (2)	0.0153 (15)	0.0165 (15)	−0.0057 (18)
C9	0.0575 (16)	0.0625 (18)	0.0699 (18)	0.0081 (14)	0.0166 (13)	0.0053 (14)
C10	0.0577 (15)	0.0411 (14)	0.0606 (16)	0.0008 (12)	0.0165 (12)	−0.0076 (12)
C11	0.0524 (14)	0.0462 (15)	0.0590 (15)	−0.0046 (12)	0.0157 (11)	−0.0034 (12)
C12	0.0420 (12)	0.0461 (14)	0.0411 (13)	0.0065 (11)	0.0038 (10)	0.0020 (11)
C13	0.0470 (13)	0.0482 (15)	0.0399 (13)	0.0018 (11)	0.0017 (10)	−0.0025 (12)
C14	0.0403 (12)	0.0489 (14)	0.0390 (12)	0.0017 (11)	0.0066 (9)	0.0001 (11)
C15	0.0441 (12)	0.0497 (14)	0.0347 (12)	0.0004 (11)	0.0077 (9)	0.0060 (11)
C16	0.0613 (16)	0.0607 (18)	0.0505 (15)	−0.0115 (13)	0.0101 (12)	−0.0079 (13)
C17	0.0453 (15)	0.098 (2)	0.0610 (17)	−0.0151 (15)	0.0092 (12)	0.0054 (17)
C18	0.0505 (15)	0.083 (2)	0.0497 (15)	0.0137 (15)	0.0154 (12)	0.0120 (15)
C19	0.0757 (18)	0.0522 (16)	0.0529 (15)	0.0090 (14)	0.0196 (13)	0.0009 (13)
C20	0.0537 (15)	0.0583 (17)	0.0487 (14)	−0.0076 (12)	0.0138 (11)	−0.0026 (13)

Geometric parameters (Å, °)

Cl1—C18	1.741 (2)	C6—H6	0.9300
S1—C14	1.661 (2)	C7—C8	1.374 (4)
O1—C13	1.212 (3)	C7—H7	0.9300
N1—C13	1.369 (3)	C8—C9	1.372 (3)
N1—C14	1.386 (3)	C8—H8	0.9300

N1—H1	0.8600	C9—H9	0.9300
N2—C14	1.329 (3)	C10—C11	1.385 (3)
N2—C15	1.430 (3)	C10—H10	0.9300
N2—H2	0.8600	C11—C12	1.387 (3)
C1—C12	1.374 (3)	C11—H11	0.9300
C1—C2	1.376 (3)	C12—C13	1.495 (3)
C1—H1A	0.9300	C15—C20	1.371 (3)
C2—C3	1.389 (3)	C15—C16	1.373 (3)
C2—H2A	0.9300	C16—C17	1.385 (3)
C3—C10	1.387 (3)	C16—H16	0.9300
C3—C4	1.487 (3)	C17—C18	1.367 (4)
C4—C5	1.375 (3)	C17—H17	0.9300
C4—C9	1.385 (3)	C18—C19	1.361 (4)
C5—C6	1.380 (3)	C19—C20	1.376 (3)
C5—H5	0.9300	C19—H19	0.9300
C6—C7	1.380 (4)	C20—H20	0.9300
C13—N1—C14	129.43 (18)	C11—C10—C3	121.2 (2)
C13—N1—H1	115.3	C11—C10—H10	119.4
C14—N1—H1	115.3	C3—C10—H10	119.4
C14—N2—C15	123.29 (19)	C10—C11—C12	119.7 (2)
C14—N2—H2	118.4	C10—C11—H11	120.2
C15—N2—H2	118.4	C12—C11—H11	120.2
C12—C1—C2	120.8 (2)	C1—C12—C11	119.4 (2)
C12—C1—H1A	119.6	C1—C12—C13	120.2 (2)
C2—C1—H1A	119.6	C11—C12—C13	120.4 (2)
C1—C2—C3	120.8 (2)	O1—C13—N1	123.8 (2)
C1—C2—H2A	119.6	O1—C13—C12	123.6 (2)
C3—C2—H2A	119.6	N1—C13—C12	112.62 (19)
C10—C3—C2	118.1 (2)	N2—C14—N1	115.62 (19)
C10—C3—C4	121.6 (2)	N2—C14—S1	125.19 (16)
C2—C3—C4	120.3 (2)	N1—C14—S1	119.17 (16)
C5—C4—C9	118.2 (2)	C20—C15—C16	120.7 (2)
C5—C4—C3	120.5 (2)	C20—C15—N2	120.8 (2)
C9—C4—C3	121.4 (2)	C16—C15—N2	118.5 (2)
C4—C5—C6	121.3 (3)	C15—C16—C17	119.2 (3)
C4—C5—H5	119.3	C15—C16—H16	120.4
C6—C5—H5	119.3	C17—C16—H16	120.4
C7—C6—C5	119.6 (3)	C18—C17—C16	119.3 (2)
C7—C6—H6	120.2	C18—C17—H17	120.3
C5—C6—H6	120.2	C16—C17—H17	120.3
C8—C7—C6	119.7 (3)	C19—C18—C17	121.6 (2)
C8—C7—H7	120.1	C19—C18—Cl1	119.0 (2)
C6—C7—H7	120.1	C17—C18—Cl1	119.4 (2)
C9—C8—C7	120.1 (3)	C18—C19—C20	119.3 (3)
C9—C8—H8	119.9	C18—C19—H19	120.4
C7—C8—H8	119.9	C20—C19—H19	120.4
C8—C9—C4	121.1 (3)	C15—C20—C19	119.9 (2)
C8—C9—H9	119.5	C15—C20—H20	120.1
C4—C9—H9	119.5	C19—C20—H20	120.1

C12—C1—C2—C3	−1.4 (3)	C14—N1—C13—O1	−2.3 (4)
C1—C2—C3—C10	2.0 (3)	C14—N1—C13—C12	176.5 (2)
C1—C2—C3—C4	−176.2 (2)	C1—C12—C13—O1	123.0 (3)
C10—C3—C4—C5	−134.7 (3)	C11—C12—C13—O1	−58.3 (3)
C2—C3—C4—C5	43.4 (3)	C1—C12—C13—N1	−55.8 (3)
C10—C3—C4—C9	45.7 (3)	C11—C12—C13—N1	122.9 (2)
C2—C3—C4—C9	−136.2 (2)	C15—N2—C14—N1	179.21 (19)
C9—C4—C5—C6	0.2 (4)	C15—N2—C14—S1	1.0 (3)
C3—C4—C5—C6	−179.4 (2)	C13—N1—C14—N2	4.0 (3)
C4—C5—C6—C7	0.4 (4)	C13—N1—C14—S1	−177.63 (18)
C5—C6—C7—C8	−1.0 (4)	C14—N2—C15—C20	63.7 (3)
C6—C7—C8—C9	1.0 (5)	C14—N2—C15—C16	−117.2 (3)
C7—C8—C9—C4	−0.4 (4)	C20—C15—C16—C17	−0.4 (4)
C5—C4—C9—C8	−0.2 (4)	N2—C15—C16—C17	−179.5 (2)
C3—C4—C9—C8	179.4 (2)	C15—C16—C17—C18	−0.5 (4)
C2—C3—C10—C11	−0.8 (4)	C16—C17—C18—C19	0.3 (4)
C4—C3—C10—C11	177.3 (2)	C16—C17—C18—Cl1	178.19 (19)
C3—C10—C11—C12	−0.9 (4)	C17—C18—C19—C20	0.7 (4)
C2—C1—C12—C11	−0.4 (3)	Cl1—C18—C19—C20	−177.18 (19)
C2—C1—C12—C13	178.3 (2)	C16—C15—C20—C19	1.4 (4)
C10—C11—C12—C1	1.6 (3)	N2—C15—C20—C19	−179.5 (2)
C10—C11—C12—C13	−177.2 (2)	C18—C19—C20—C15	−1.6 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.86	2.00	2.683 (3)	135
N1—H1···S1^{i}	0.86	2.55	3.362 (3)	157
N2—H2···O1^{ii}	0.86	2.58	3.210 (3)	131
C1—H1A···Cg3^{ii}	0.93	2.98	3.586 (3)	124

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) −*x*+2, *y*, −*z*+1/2.

Fig. 1

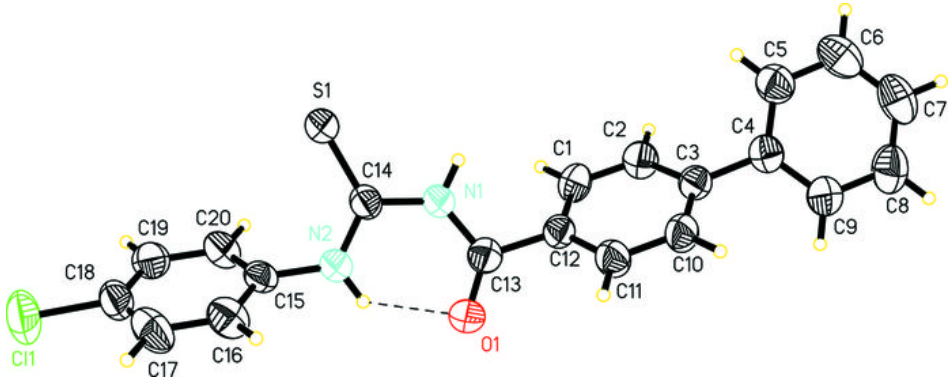
S1
C14
N1
C1
C2
C3
C4
C5
C6
C7
C8
C9
C10
C11
C12
C13
N2
C15
C16
C17
C18
C19
C20
Cl1
O1

Fig. 2