Bis(N-phenylethane-1,2-diamine)dithio-cyanatonickel(II)

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The asymmetric unit of the title mononuclear Ni^{II} compound, $[Ni(NCS)_2(C_8H_{12}N_2)_2]$, contains two independent half-molecules, the Ni atoms of which lie on crystallographic inversion centres. Each Ni^{II} ion is chelated by two N atoms from two N-phenylethane-1,2-diamine ligands and is also coordinated by two N atoms from two thiocyanate ligands, giving a distorted octahedral geometry. In the crystal, molecules are linked into a two-dimensional network parallel to (100) by N—H···S interactions.

Related literature

For related structures, see: Lever *et al.* (1983); Brown & Lingafelter (1963); Sanni *et al.* (1987).
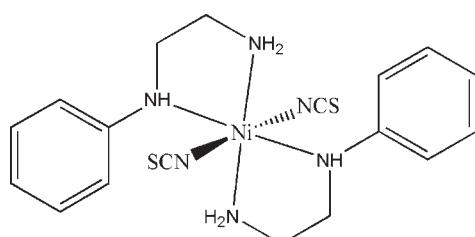
Experimental

Crystal data

$[Ni(NCS)_2(C_8H_{12}N_2)_2]$
$M_r = 447.26$
Triclinic, $P\bar{1}$
$a = 7.9947\ (2)$ Å
$b = 9.4708\ (3)$ Å
$c = 13.8044\ (3)$ Å
$\alpha = 93.045\ (1)°$
$\beta = 98.258\ (1)°$

$\gamma = 90.934\ (1)°$
$V = 1032.62\ (5)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 1.16$ mm^{-1}
$T = 298$ K
$0.18 \times 0.17 \times 0.17$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.819$, $T_{max} = 0.828$

6197 measured reflections
4314 independent reflections
3284 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.080$
$S = 1.03$
4314 reflections
253 parameters
2 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—N5	2.073 (2)	Ni2—N6	2.047 (2)
Ni1—N1	2.094 (2)	Ni2—N3	2.104 (2)
Ni1—N2	2.159 (2)	Ni2—N4	2.171 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···S1^i	0.89 (1)	2.52 (1)	3.393 (2)	168 (3)
N2—H2···S2^{ii}	0.90 (1)	2.67 (2)	3.436 (2)	144 (3)

Symmetry codes: (i) $x + 1, y - 1, z$; (ii) $x - 1, y, z$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5008).

References

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supplementary materials

Bis(*N*-phenylethane-1,2-diamine)dithiocyanatonickel(II)

C.-Y. Wang, F. Cao, P. Wang, C.-Y. Lv and X. Wu

Comment

As part of our investigations into novel urease inhibitors, we have synthesized the title compound, a new Ni^{II} complex. There are two independent half-molecules in the asymmetric unit. Each Ni atom lies on an inversion centre and is chelated by two N atoms from two *N*-phenylethane-1,2-diamine ligands, and coordinated by two N atoms from two thiocyanate ligands (Fig. 1). While the three *trans* angles at each Ni centre are 180° by symmetry, the other angles are close to 90° [81.54 (8)°–98.46 (8)°], indicating a slightly distorted octahedral coordination. The Ni—N bond lengths (Table 1) are typical and are comparable with those observed in other similar nickel(II) complexes (Lever *et al.*, 1983; Brown & Lingafelter, 1963; Sanni *et al.*, 1987).

Experimental

N-Phenylethane-1,2-diamine (0.2 mmol, 27.2 mg), ammonium thiocyanate (0.2 mmol, 15.2 mg), and $Ni(CH_3COO)_2.4H_2O$ (0.1 mmol, 24.9 mg) were mixed in a MeOH solution with stirring for 30 min at room temperature. After keeping the filtrate in air for five days, green block-shaped crystals were formed at the bottom of the vessel.

Refinement

Atoms H2 and H4A were located in a difference Fourier map and refined isotropically, with N-H distances restrained to 0.90 (1) Å. Other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C-H distances in the range 0.93–0.97 Å, N–H distances of 0.90 Å, and with $U_{iso}(H)$ set at $1.2U_{eq}(C,N)$.

Figures
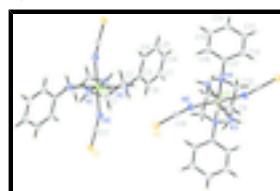
Fig. 1. View of the two independent half-molecules in the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Unlabelled atoms in the molecule containing Ni1 are at the symmetry position (-*x*, 2 - *y*, -*z*) and those in the other molecule are at (2 -*x*, 1 - *y*, 1 -*z*).

Bis(*N*-phenylethane-1,2-diamine)dithiocyanatonickel(II)

Crystal data

$[Ni(NCS)_2(C_8H_{12}N_2)_2]$	$Z = 2$
$M_r = 447.26$	$F(000) = 468$
Triclinic, $P\bar{1}$	$D_x = 1.438$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

$a = 7.9947$ (2) Å

$b = 9.4708$ (3) Å

$c = 13.8044$ (3) Å

$\alpha = 93.045$ (1)°

$\beta = 98.258$ (1)°

$\gamma = 90.934$ (1)°

$V = 1032.62$ (5) Å^3

Cell parameters from 1847 reflections

$\theta = 2.5–25.0°$

$\mu = 1.16$ mm^{-1}

$T = 298$ K

Block, green

$0.18 \times 0.17 \times 0.17$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

graphite

ω scan

Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)

$T_{min} = 0.819$, $T_{max} = 0.828$

6197 measured reflections

4314 independent reflections

3284 reflections with $I > 2\sigma(I)$

$R_{int} = 0.018$

$\theta_{max} = 27.0°$, $\theta_{min} = 1.5°$

$h = -9 \rightarrow 9$

$k = -12 \rightarrow 8$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.080$

$S = 1.03$

4314 reflections

253 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0256P)^2 + 0.3717P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.31$ e Å^{-3}

$\Delta\rho_{min} = -0.26$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Ni1	0.0000	1.0000	0.0000	0.03175 (12)
Ni2	1.0000	0.5000	0.5000	0.03243 (12)
S1	0.21085 (10)	1.40198 (7)	0.20456 (5)	0.0487 (2)
S2	1.28479 (10)	0.83224 (9)	0.32626 (6)	0.0600 (2)
N1	0.2337 (3)	0.9430 (2)	−0.04011 (16)	0.0424 (5)
H1A	0.2188	0.9022	−0.1012	0.051*
H1B	0.3009	1.0204	−0.0392	0.051*
N2	0.0504 (3)	0.8295 (2)	0.09707 (15)	0.0348 (5)
N3	1.2463 (3)	0.4484 (2)	0.56125 (16)	0.0444 (6)
H3A	1.3139	0.5263	0.5682	0.053*
H3B	1.2448	0.4147	0.6209	0.053*
N4	1.0325 (3)	0.3174 (2)	0.40317 (16)	0.0372 (5)
N5	0.1256 (3)	1.1389 (2)	0.10846 (17)	0.0460 (6)
N6	1.1013 (3)	0.6271 (2)	0.40688 (16)	0.0435 (6)
C1	0.3124 (3)	0.8433 (3)	0.0301 (2)	0.0452 (7)
H1C	0.3664	0.8947	0.0893	0.054*
H1D	0.3979	0.7902	0.0017	0.054*
C2	0.1773 (3)	0.7435 (3)	0.0544 (2)	0.0411 (6)
H2A	0.1247	0.6904	−0.0044	0.049*
H2B	0.2265	0.6772	0.1010	0.049*
C3	−0.0938 (3)	0.7571 (2)	0.12332 (18)	0.0345 (6)
C4	−0.1649 (3)	0.6357 (3)	0.0722 (2)	0.0419 (6)
H4	−0.1159	0.5953	0.0205	0.050*
C5	−0.3091 (4)	0.5748 (3)	0.0985 (2)	0.0514 (8)
H5	−0.3563	0.4933	0.0640	0.062*
C6	−0.3842 (4)	0.6325 (3)	0.1746 (2)	0.0541 (8)
H6	−0.4811	0.5906	0.1916	0.065*
C7	−0.3129 (4)	0.7538 (3)	0.2254 (2)	0.0498 (7)
H7	−0.3627	0.7941	0.2768	0.060*
C8	−0.1695 (3)	0.8152 (3)	0.2006 (2)	0.0427 (6)
H8	−0.1224	0.8963	0.2356	0.051*
C9	1.3121 (3)	0.3404 (3)	0.4961 (2)	0.0501 (7)
H9A	1.4042	0.2912	0.5325	0.060*
H9B	1.3546	0.3851	0.4426	0.060*
C10	1.1697 (3)	0.2371 (3)	0.4563 (2)	0.0486 (7)
H10A	1.2089	0.1657	0.4124	0.058*
H10B	1.1293	0.1902	0.5096	0.058*
C11	0.8788 (3)	0.2437 (3)	0.36351 (19)	0.0369 (6)
C12	0.8105 (4)	0.1359 (3)	0.4109 (2)	0.0448 (7)
H12	0.8716	0.1011	0.4667	0.054*
C13	0.6509 (4)	0.0801 (3)	0.3749 (2)	0.0532 (8)
H13	0.6059	0.0080	0.4071	0.064*
C14	0.5582 (4)	0.1295 (3)	0.2925 (2)	0.0551 (8)
H14	0.4505	0.0923	0.2697	0.066*
C15	0.6271 (4)	0.2353 (3)	0.2438 (2)	0.0492 (7)

H15	0.5663	0.2686	0.1874	0.059*
C16	0.7864 (3)	0.2914 (3)	0.2791 (2)	0.0428 (6)
H16	0.8321	0.3621	0.2459	0.051*
C17	0.1622 (3)	1.2472 (3)	0.14957 (18)	0.0353 (6)
C18	1.1772 (3)	0.7109 (3)	0.37193 (18)	0.0372 (6)
H2	0.104 (4)	0.874 (3)	0.1524 (14)	0.080*
H4A	1.076 (4)	0.353 (3)	0.3537 (17)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0354 (3)	0.0256 (2)	0.0348 (3)	−0.00085 (18)	0.00618 (19)	0.00362 (18)
Ni2	0.0350 (3)	0.0293 (2)	0.0333 (3)	−0.00465 (19)	0.00483 (19)	0.00667 (19)
S1	0.0621 (5)	0.0367 (4)	0.0477 (4)	−0.0126 (3)	0.0130 (4)	−0.0036 (3)
S2	0.0544 (5)	0.0756 (6)	0.0502 (5)	−0.0305 (4)	0.0059 (4)	0.0199 (4)
N1	0.0442 (13)	0.0376 (12)	0.0484 (14)	0.0029 (10)	0.0135 (11)	0.0114 (10)
N2	0.0373 (12)	0.0311 (11)	0.0371 (13)	−0.0011 (9)	0.0079 (10)	0.0054 (9)
N3	0.0426 (13)	0.0445 (13)	0.0439 (14)	−0.0035 (10)	−0.0003 (10)	0.0028 (11)
N4	0.0372 (12)	0.0370 (12)	0.0374 (13)	−0.0042 (10)	0.0050 (10)	0.0053 (10)
N5	0.0558 (15)	0.0365 (13)	0.0436 (14)	−0.0056 (11)	0.0019 (11)	0.0002 (11)
N6	0.0518 (15)	0.0391 (13)	0.0415 (13)	−0.0066 (11)	0.0126 (11)	0.0075 (10)
C1	0.0382 (16)	0.0445 (16)	0.0552 (18)	0.0092 (12)	0.0096 (13)	0.0145 (13)
C2	0.0434 (16)	0.0325 (14)	0.0494 (17)	0.0092 (12)	0.0090 (13)	0.0120 (12)
C3	0.0353 (14)	0.0312 (13)	0.0375 (14)	0.0001 (11)	0.0026 (11)	0.0132 (11)
C4	0.0494 (17)	0.0345 (14)	0.0419 (16)	0.0006 (12)	0.0049 (13)	0.0080 (12)
C5	0.0481 (18)	0.0373 (16)	0.067 (2)	−0.0073 (13)	−0.0003 (15)	0.0110 (14)
C6	0.0368 (16)	0.0563 (19)	0.071 (2)	−0.0027 (14)	0.0066 (15)	0.0288 (17)
C7	0.0505 (18)	0.0519 (18)	0.0511 (18)	0.0062 (14)	0.0161 (14)	0.0153 (14)
C8	0.0485 (17)	0.0408 (15)	0.0411 (16)	0.0003 (12)	0.0124 (13)	0.0089 (12)
C9	0.0384 (16)	0.0549 (18)	0.0569 (19)	0.0059 (13)	0.0047 (14)	0.0046 (15)
C10	0.0473 (17)	0.0423 (16)	0.0556 (19)	0.0067 (13)	0.0061 (14)	−0.0004 (14)
C11	0.0413 (15)	0.0308 (13)	0.0386 (15)	−0.0011 (11)	0.0084 (12)	−0.0030 (11)
C12	0.0545 (18)	0.0361 (15)	0.0440 (16)	−0.0061 (13)	0.0080 (13)	0.0040 (12)
C13	0.061 (2)	0.0409 (16)	0.059 (2)	−0.0167 (14)	0.0143 (16)	0.0009 (14)
C14	0.0462 (18)	0.0518 (18)	0.065 (2)	−0.0127 (14)	0.0053 (15)	−0.0053 (16)
C15	0.0478 (18)	0.0529 (18)	0.0453 (17)	0.0017 (14)	0.0027 (13)	−0.0008 (14)
C16	0.0440 (16)	0.0426 (15)	0.0422 (16)	−0.0039 (12)	0.0070 (13)	0.0059 (12)
C17	0.0330 (14)	0.0420 (15)	0.0318 (14)	−0.0013 (11)	0.0054 (11)	0.0091 (12)
C18	0.0376 (15)	0.0402 (15)	0.0335 (14)	−0.0013 (12)	0.0048 (11)	0.0014 (11)

Geometric parameters (Å, °)

Ni1—N5	2.073 (2)	C1—H1D	0.97
Ni1—N5^i	2.073 (2)	C2—H2A	0.97
Ni1—N1^i	2.094 (2)	C2—H2B	0.97
Ni1—N1	2.094 (2)	C3—C4	1.386 (3)
Ni1—N2^i	2.159 (2)	C3—C8	1.393 (4)
Ni1—N2	2.159 (2)	C4—C5	1.384 (4)

Ni2—N6[ii]	2.047 (2)	C4—H4	0.93
Ni2—N6	2.047 (2)	C5—C6	1.376 (4)
Ni2—N3[ii]	2.104 (2)	C5—H5	0.93
Ni2—N3	2.104 (2)	C6—C7	1.384 (4)
Ni2—N4[ii]	2.171 (2)	C6—H6	0.93
Ni2—N4	2.171 (2)	C7—C8	1.372 (4)
S1—C17	1.629 (3)	C7—H7	0.93
S2—C18	1.630 (3)	C8—H8	0.93
N1—C1	1.473 (3)	C9—C10	1.511 (4)
N1—H1A	0.90	C9—H9A	0.97
N1—H1B	0.90	C9—H9B	0.97
N2—C3	1.432 (3)	C10—H10A	0.97
N2—C2	1.479 (3)	C10—H10B	0.97
N2—H2	0.898 (10)	C11—C12	1.387 (3)
N3—C9	1.479 (3)	C11—C16	1.388 (4)
N3—H3A	0.90	C12—C13	1.386 (4)
N3—H3B	0.90	C12—H12	0.93
N4—C11	1.426 (3)	C13—C14	1.374 (4)
N4—C10	1.475 (3)	C13—H13	0.93
N4—H4A	0.889 (10)	C14—C15	1.386 (4)
N5—C17	1.158 (3)	C14—H14	0.93
N6—C18	1.158 (3)	C15—C16	1.383 (4)
C1—C2	1.508 (3)	C15—H15	0.93
C1—H1C	0.97	C16—H16	0.93
N5—Ni1—N5[i]	180	N1—C1—H1D	109.9
N5—Ni1—N1[i]	90.87 (9)	C2—C1—H1D	109.9
N5[i]—Ni1—N1[i]	89.13 (9)	H1C—C1—H1D	108.3
N5—Ni1—N1	89.13 (9)	N2—C2—C1	107.7 (2)
N5[i]—Ni1—N1	90.87 (9)	N2—C2—H2A	110.2
N1[i]—Ni1—N1	180	C1—C2—H2A	110.2
N5—Ni1—N2[i]	90.77 (8)	N2—C2—H2B	110.2
N5[i]—Ni1—N2[i]	89.23 (8)	C1—C2—H2B	110.2
N1[i]—Ni1—N2[i]	82.40 (8)	H2A—C2—H2B	108.5
N1—Ni1—N2[i]	97.60 (8)	C4—C3—C8	119.0 (2)
N5—Ni1—N2	89.23 (8)	C4—C3—N2	122.6 (2)
N5[i]—Ni1—N2	90.77 (8)	C8—C3—N2	118.3 (2)
N1[i]—Ni1—N2	97.60 (8)	C5—C4—C3	119.6 (3)
N1—Ni1—N2	82.40 (8)	C5—C4—H4	120.2
N2[i]—Ni1—N2	180	C3—C4—H4	120.2
N6[ii]—Ni2—N6	180	C6—C5—C4	121.3 (3)
N6[ii]—Ni2—N3[ii]	89.13 (9)	C6—C5—H5	119.3
N6—Ni2—N3[ii]	90.87 (9)	C4—C5—H5	119.3
N6[ii]—Ni2—N3	90.87 (9)	C5—C6—C7	118.8 (3)
N6—Ni2—N3	89.13 (9)	C5—C6—H6	120.6
N3[ii]—Ni2—N3	180	C7—C6—H6	120.6

N6[ii]—Ni2—N4[ii]	89.54 (8)	C8—C7—C6	120.6 (3)
N6—Ni2—N4[ii]	90.46 (8)	C8—C7—H7	119.7
N3[ii]—Ni2—N4[ii]	81.54 (8)	C6—C7—H7	119.7
N3—Ni2—N4[ii]	98.46 (8)	C7—C8—C3	120.6 (3)
N6[ii]—Ni2—N4	90.46 (8)	C7—C8—H8	119.7
N6—Ni2—N4	89.54 (8)	C3—C8—H8	119.7
N3[ii]—Ni2—N4	98.46 (8)	N3—C9—C10	108.3 (2)
N3—Ni2—N4	81.54 (8)	N3—C9—H9A	110.0
N4[ii]—Ni2—N4	180	C10—C9—H9A	110.0
C1—N1—Ni1	108.38 (16)	N3—C9—H9B	110.0
C1—N1—H1A	110.0	C10—C9—H9B	110.0
Ni1—N1—H1A	110.0	H9A—C9—H9B	108.4
C1—N1—H1B	110.0	N4—C10—C9	107.8 (2)
Ni1—N1—H1B	110.0	N4—C10—H10A	110.1
H1A—N1—H1B	108.4	C9—C10—H10A	110.1
C3—N2—C2	117.70 (19)	N4—C10—H10B	110.1
C3—N2—Ni1	116.59 (15)	C9—C10—H10B	110.1
C2—N2—Ni1	104.74 (15)	H10A—C10—H10B	108.5
C3—N2—H2	107 (2)	C12—C11—C16	118.8 (2)
C2—N2—H2	107 (2)	C12—C11—N4	122.6 (2)
Ni1—N2—H2	103 (2)	C16—C11—N4	118.2 (2)
C9—N3—Ni2	109.24 (16)	C13—C12—C11	119.9 (3)
C9—N3—H3A	109.8	C13—C12—H12	120.1
Ni2—N3—H3A	109.8	C11—C12—H12	120.1
C9—N3—H3B	109.8	C14—C13—C12	121.2 (3)
Ni2—N3—H3B	109.8	C14—C13—H13	119.4
H3A—N3—H3B	108.3	C12—C13—H13	119.4
C11—N4—C10	118.3 (2)	C13—C14—C15	119.2 (3)
C11—N4—Ni2	114.31 (16)	C13—C14—H14	120.4
C10—N4—Ni2	105.40 (16)	C15—C14—H14	120.4
C11—N4—H4A	108 (2)	C16—C15—C14	120.0 (3)
C10—N4—H4A	105 (2)	C16—C15—H15	120.0
Ni2—N4—H4A	104 (2)	C14—C15—H15	120.0
C17—N5—Ni1	156.7 (2)	C15—C16—C11	120.9 (3)
C18—N6—Ni2	165.9 (2)	C15—C16—H16	119.6
N1—C1—C2	108.8 (2)	C11—C16—H16	119.6
N1—C1—H1C	109.9	N5—C17—S1	178.3 (3)
C2—C1—H1C	109.9	N6—C18—S2	178.0 (2)

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

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···S1[iii]	0.89 (1)	2.52 (1)	3.393 (2)	168 (3)
N2—H2···S2[iv]	0.90 (1)	2.67 (2)	3.436 (2)	144 (3)

Symmetry codes: (iii) x+1, y−1, z; (iv) x−1, y, z.

Fig. 1