Discussion. The structure consists of discrete dinuclear molecules linked by van der Waals forces. A view of the molecule with the numbering of atoms is shown in Fig. 1. Atom parameters are in Table 1* and selected bond distances and angles are in Table 2. The molecule contains two organic bridges: 1-phenyl-1-propenyl and ethoxymethylidyne groups. The first is σ bonded to Fe(2) with an Fe(2)—C(4) distance of 2·014 (10) Å and unsymmetrically π bonded to Fe(1) with Fe(1)—C(4) 0·089 (21) Å shorter than Fe(1)—C(5), as is usual in other known complexes of this type (Iggo, Mays, Raithby & Hendrick, 1983 and references therein). The phenyl and methyl groups are in *cis* configuration, forming a torsion angle of −15·4 (9)°, similar to that observed in the 1,2-diphenylvinyl complex (Ros *et al.*, 1985), where this angle is −13·1°. The substitution of one phenyl by a methyl group in this ligand produces a shortening of the C(4)—C(5) [1·40 (1) and 1·47 (3) Å in the 1-phenyl-1-propenyl and 1,2-diphenylvinyl complexes, respectively] and Fe(1)—C(4) bond distances [2·092 (10) and 2·15 (2) Å, respectively], giving the ligand a geometry intermediate between that in the 1,2-diphenylvinyl complex and that in $Fe_2(CO)_5\{\mu\text{-}C(OC_2H_5)C[C(O)OCH_3]C[C(O)OCH_3]\}\{\mu\text{-}C(C_6H_5)\text{-}C(C_6H_5)H\}$ (Ros, Commenges, Mathieu, Solans & Font-Altaba, 1984).

The bridging ethoxymethylidyne group is bonded almost symmetrically to Fe(1) and Fe(2) and is situated on the opposite side to the vinyl group, minimizing interactions, as in the 1,2-diphenylvinyl complex.

The Fe—Fe bond length of 2·549 (2) Å is close to the Fe—Fe distances found for most structures containing the $Fe_2(CO)_6$ moiety (Krüger, Barnett & Brauer, 1978) and the bond distances and angles of each $Fe(CO)_3$ moiety have values typical of those reported in the literature.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and full bond length and angle data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42232 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Structures of 2′-[1-(2-Pyridyl)ethylidene]-3-azabicyclo[3.2.2]nonane-3-carbothiohydrazide, $C_{16}H_{22}N_4S$, and -3-carboselenohydrazide, $C_{16}H_{22}N_4Se$*

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Abstract. $C_{16}H_{22}N_4S$ (I): $M_r = 302{\cdot}4$, monoclinic, Cc, $a = 6{\cdot}433$ (1), $b = 21{\cdot}796$ (3), $c = 11{\cdot}533$ (2) Å, $\beta = 90{\cdot}48$ (1)°, $V = 1616{\cdot}9$ Å^3, $Z = 4$, D_m(flotation) = 1·23 (1), $D_x = 1{\cdot}243$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 1{\cdot}297$ mm^{-1}, $F(000) = 648{\cdot}0$, $T = 293$ K, $R = 0{\cdot}049$ for 991 observed reflections. $C_{16}H_{22}N_4Se$ (II): $M_r = 349{\cdot}3$, triclinic, $P\bar{1}$, $a = 8{\cdot}462$ (1), $b = 9{\cdot}178$ (1),

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$c = 11{\cdot}128\,(1)$ Å, $\alpha = 107{\cdot}56\,(1)$, $\beta = 95{\cdot}07\,(1)$, $\gamma = 86{\cdot}86\,(1)°$, $V = 820{\cdot}4$ Å^3, $Z = 2$, D_m(flotation) = $1{\cdot}40\,(1)$, $D_x = 1{\cdot}414$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 3{\cdot}914$ mm^{-1}, $F(000) = 360{\cdot}0$, $T = 293$ K, $R = 0{\cdot}039$ for 1821 observed reflections. The azabicyclo[3.2.2]nonane ring adopts the boat–boat conformation in both compounds. The C—S and C—Se bond lengths are intermediate between single and double bonds, as found in thiosemicarbazones. The C—N(aza) bond in both compounds also has partial double-bond character. There is an intramolecular H bond of the type N—H···S (Se) with N···S 2·78 (1) Å and N···Se 2·933 (5) Å.

Table 1. *Fractional atomic coordinates* (×10^4) *with e.s.d.'s in parentheses and equivalent isotropic temperature factors for* (I)

$$B_{\rm eq} = \tfrac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{13}ac\cos\beta).$$

	x	y	z	$B_{\rm eq}$(Å^2)
C(1)	4920 (14)	1401 (4)	2347 (8)	3·65
C(2)	7798 (14)	435 (4)	575 (8)	3·83
C(3)	9701 (15)	88 (4)	873 (8)	3·94
C(4)	2376 (18)	2021 (5)	3387 (10)	5·30
C(5)	2088 (15)	2723 (4)	3304 (9)	4·47
C(6)	−90 (17)	2900 (5)	2851 (9)	5·03
C(7)	−438 (18)	2740 (5)	1596 (11)	5·81
C(8)	1495 (15)	2490 (5)	980 (8)	4·87
C(9)	1930 (20)	1824 (6)	1271 (11)	7·07
C(10)	3390 (20)	2888 (6)	1224 (11)	6·87
C(11)	3792 (18)	3001 (5)	2521 (10)	5·21
C(12)	12273 (16)	−83 (5)	2168 (8)	6·91
C(13)	13280 (15)	−480 (4)	1462 (8)	6·14
C(14)	12379 (15)	−600 (4)	401 (9)	5·90
C(15)	10546 (14)	−310 (4)	75 (8)	5·17
C(16)	6748 (14)	427 (4)	−597 (7)	5·62
N(1)	3184 (10)	1737 (3)	2340 (5)	4·56
N(2)	5324 (10)	1125 (3)	1309 (6)	4·34
N(3)	7056 (9)	777 (3)	1406 (5)	3·99
N(4)	10521 (11)	202 (3)	1897 (6)	5·46
S	6437 (5)	1318 (1)	3561 (3)	4·07

Table 2. *Fractional atomic coordinates* (×10^4) *with e.s.d.'s in parentheses and equivalent isotropic temperature factors for* (II)

$$B_{\rm eq} = \tfrac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}ab + \beta_{13}ac + \beta_{23}bc)$$

	x	y	z	$B_{\rm eq}$(Å^2)
C(1)	3054 (6)	10679 (5)	2754 (4)	3·23
C(2)	6446 (5)	9610 (5)	1121 (4)	3·16
C(3)	6975 (6)	8144 (5)	249 (4)	3·38
C(4)	3400 (7)	13244 (6)	4247 (6)	5·02
C(5)	3741 (7)	13552 (6)	5678 (5)	4·67
C(6)	2459 (9)	14540 (7)	6441 (6)	6·10
C(7)	948 (8)	13677 (7)	6392 (6)	6·04
C(8)	1008 (6)	12044 (6)	5523 (5)	4·02
C(9)	924 (6)	11903 (6)	4127 (5)	3·94
C(10)	2462 (7)	11201 (6)	5916 (5)	4·94
C(11)	3994 (8)	12077 (7)	6040 (7)	6·13
C(12)	8838 (7)	6580 (6)	−1084 (6)	5·17
C(13)	7850 (8)	5404 (6)	−1318 (6)	6·13
C(14)	6455 (9)	5647 (7)	−755 (7)	6·51
C(15)	6010 (6)	6976 (5)	28 (5)	4·21
C(16)	7406 (6)	10999 (6)	1477 (5)	4·47
N(1)	2460 (5)	11887 (4)	3644 (4)	3·99
N(2)	4481 (5)	10880 (4)	2423 (3)	3·66
N(3)	5080 (5)	9624 (4)	1569 (3)	3·47
N(4)	8412 (6)	8002 (6)	−272 (5)	6·12
Se	1904 (1)	8884 (1)	2031 (1)	4·05

Introduction. Thio- and selenosemicarbazones belong to a special class of chelating molecules, which possess a wide range of medicinal properties. The thio derivative of the title compound (I) has been found to be the most potent anti-malarial drug and also possesses antileukaemic properties. Both derivatives were crystallized from methyl alcohol (Bhoon, Scovill & Klayman, 1983).

Experimental. $C_{16}H_{22}N_4S$ (I): crystal ~0·20 × 0·25 × 0·60 mm. Nonius CAD-4F-11M diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, scan speed 1° min^{-1}, $\theta < 24°$, h 0 to 7, k 0 to 24, l −13 to 13. 1485 reflections collected, 991 judged significant ($|F_o| > 3\sigma|F_o|$), lattice parameters from 21 reflections ($14 < 2\theta < 32°$), three standard reflections ($\bar{2}2\bar{1}$, 004 and $\bar{1}\bar{9}3$) every 2000 s, 4% variation in intensity. No corrections for absorption. Structure solved by direct methods, *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement (on F) of scale factor, positional and anisotropic thermal parameters (isotropic thermal parameters for H atoms, initial H positions calculated by stereochemistry) converged to $R = 0{\cdot}049$, $wR = 0{\cdot}046$, $S = 2{\cdot}45$; $w(|F_o| - |F_c|)^2$ minimized, $w = (9{\cdot}0 + 1{\cdot}0|F_o| + 0{\cdot}016|F_o|^2)^{-1}$. $(\Delta/\sigma)_{\rm max} = 0{\cdot}1$. Final $\Delta\rho$ excursions $< |0{\cdot}3|$ e Å^{-3}. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). *LALS* (Gantzel, Sparks & Trueblood, 1961) for refinement.

$C_{16}H_{22}N_4Se$ (II): crystal ~0·15 × 0·25 × 0·70 mm, $\theta < 23{\cdot}5°$, h 0 to 9, k −10 to 10, l −12 to 12. 2747 reflections collected, 1821 judged significant ($|F_o| > 3\sigma|F_o|$), lattice parameters from 22 reflections ($16 < 2\theta < 32°$), three standard reflections ($\bar{3}30$, $0\bar{5}0$ and 005) every 2000 s, 4% variation in intensity. Structure solved by direct methods, *MULTAN*78. Full-matrix least-squares refinement as above converged to $R = 0{\cdot}039$, $wR = 0{\cdot}038$, $S = 1{\cdot}05$; $w(|F_o| - |F_c|)^2$ minimized, $w = (6{\cdot}0 + 1{\cdot}0|F_o| + 0{\cdot}015|F_o|^2)^{-1}$. $(\Delta/\sigma)_{\rm max} = 0{\cdot}1$. Final $\Delta\rho$ excursions $< |0{\cdot}3|$ e Å^{-3}. No correction for secondary extinction.

Discussion. The atomic parameters with their e.s.d.'s and equivalent isotropic temperature factors are given in Tables 1 and 2 for the thio and seleno derivatives. Bond lengths and bond angles involving the non-hydrogen atoms for both derivatives are given in Table 3. The chemical formula with atomic numbering is given in Fig. 1. A perspective view of (I) is shown in Fig. 2.*

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a perspective view of (II) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42100 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The C—S bond distance in (I) is 1·71 (1) Å, which agrees very well with C—S distances in thiosemicarbazones (Restivo & Palenik, 1970). The C—S distance in all these cases is intermediate between the C—S single-bond distance of 1·82 Å and the double-bond value of 1·56 Å (Sutton, 1965), suggesting partial double-bond character. Similarly, the C—Se bond in (II) is 1·883 (5) Å while the C—Se single-bond value is 1·92 Å and the double-bond value is 1·71 Å (Conde, López-Castro & Márquez, 1972). The C(2)—N(3) bond distances in (I) and (II) of 1·31 (1) and 1·297 (6) Å, respectively, indicate double-bond character, while the C(1)—N(1) bond distances of 1·34 (1) and 1·352 (6) Å and C(1)—N(2) bond distances of 1·37 (1) and 1·331 (7) Å in (I) and (II) indicate partial double-bond character. There is an intramolecular hydrogen bond between N(3) and S in (I) and N(3) and Se in (II): N(3)···S = 2·78 (1) Å, N(3)—H···S = 122 (1), S···H = 2·17 (7) Å; N(3)···Se = 2·933 (5) Å, N(3)—H···Se = 133 (1)°, Se···H = 2·12 (5) Å.

In both (I) and (II) the azabicyclo[3.2.2]nonane ring can be regarded as formed by the fusion of the cycloheptane ring *L* and cyclohexane ring *R* (Table 4).

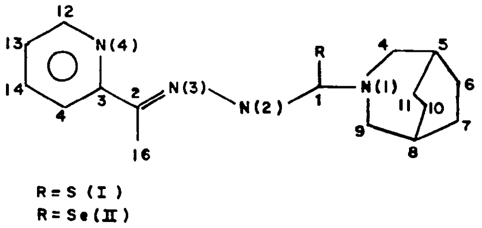

Fig. 1. Crystallographic numbering of atoms.

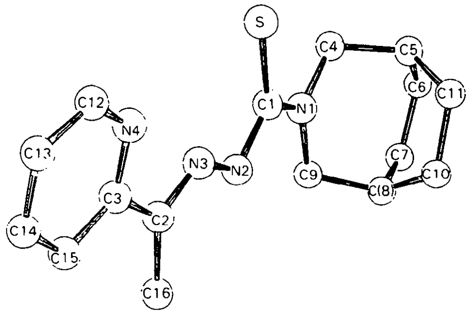

Fig. 2. Perspective view of molecule (I).

Table 3. *Intramolecular bond lengths* (Å) *and angles* (°) *and their e.s.d.'s* (*in parentheses*) *for* (I) *and* (II)

R = S in (I), Se in (II).

	(I)	(II)		(I)	(II)
C(1)—*R*	1·71 (1)	1·883 (5)	C(1)—N(1)	1·34 (1)	1·352 (6)
C(1)—N(2)	1·37 (1)	1·331 (7)	C(2)—N(3)	1·31 (1)	1·297 (6)
C(3)—N(4)	1·31 (1)	1·376 (7)	C(4)—N(1)	1·46 (1)	1·464 (7)
C(9)—N(1)	1·48 (1)	1·448 (7)	C(12)—N(4)	1·32 (1)	1·394 (8)
N(2)—N(3)	1·352 (9)	1·360 (5)	C(2)—C(3)	1·48 (1)	1·475 (6)
C(2)—C(16)	1·51 (1)	1·481 (7)	C(3)—C(15)	1·38 (1)	1·333 (7)
C(4)—C(5)	1·54 (1)	1·534 (8)	C(5)—C(6)	1·54 (1)	1·520 (9)
C(5)—C(11)	1·55 (2)	1·525 (9)	C(6)—C(7)	1·50 (2)	1·529 (10)
C(7)—C(8)	1·54 (2)	1·520 (9)	C(8)—C(9)	1·52 (2)	1·515 (8)
C(8)—C(10)	1·52 (2)	1·519 (8)	C(10)—C(11)	1·54 (2)	1·536 (9)
C(12)—C(13)	1·36 (1)	1·350 (9)	C(13)—C(14)	1·37 (1)	1·362 (10)
C(14)—C(15)	1·39 (1)	1·325 (9)			

	(I)	(II)
N(1)—C(1)—N(2)	113·7 (7)	114·7 (4)
N(2)—C(1)—*R*	124·0 (7)	123·5 (3)
C(3)—C(2)—N(3)	115·4 (8)	116·2 (4)
C(2)—C(3)—C(15)	119·8 (8)	116·4 (4)
C(15)—C(3)—N(4)	124·2 (8)	122·7 (5)
C(4)—C(5)—C(6)	112·2 (8)	113·1 (5)
C(6)—C(5)—C(11)	110·5 (8)	109·3 (5)
C(6)—C(7)—C(8)	114·4 (9)	113·1 (5)
C(7)—C(8)—C(10)	112·3 (9)	109·5 (5)
C(8)—C(9)—N(1)	113·9 (9)	113·7 (4)
C(5)—C(11)—C(10)	113·0 (9)	113·4 (5)
C(12)—C(13)—C(14)	117·1 (9)	118·7 (6)
C(3)—C(15)—C(14)	116·4 (8)	117·6 (5)
C(1)—N(1)—C(9)	121·7 (8)	123·4 (4)
C(1)—N(2)—N(3)	109·7 (6)	113·5 (4)
C(2)—N(3)—N(2)	124·4 (7)	122·3 (4)
N(1)—C(1)—*R*	122·3 (7)	121·7 (3)
C(3)—C(2)—C(16)	124·6 (8)	122·6 (4)
C(16)—C(2)—N(3)	120·0 (8)	121·2 (4)
C(2)—C(3)—N(4)	116·0 (8)	120·9 (4)
C(5)—C(4)—N(1)	114·4 (8)	113·6 (5)
C(4)—C(5)—C(11)	109·8 (8)	112·0 (5)
C(5)—C(6)—C(7)	113·3 (9)	113·2 (5)
C(7)—C(8)—C(9)	112·7 (9)	114·6 (5)
C(9)—C(8)—C(10)	111·1 (9)	111·6 (4)
C(8)—C(10)—C(11)	113·6 (10)	112·5 (5)
C(13)—C(12)—N(4)	124·6 (9)	119·5 (6)
C(13)—C(14)—C(15)	120·4 (9)	123·7 (6)
C(1)—N(1)—C(4)	122·2 (7)	121·1 (4)
C(4)—N(1)—C(9)	116·1 (8)	115·4 (4)
C(3)—N(4)—C(12)	117·3 (8)	117·7 (5)

Table 4. *Torsion angles* (°) *for* (I) *and* (II)

	(I)	(II)
Ring *B* [atoms N(1), C(4), C(5), C(6), C(7), C(8), C(9)]		
C(5)—C(4)—N(1)—C(9)	−59 (1)	58·4 (6)
N(1)—C(4)—C(5)—C(6)	90 (1)	−88·6 (6)
C(4)—C(5)—C(6)—C(7)	−69 (1)	75·1 (7)
C(5)—C(6)—C(7)—C(8)	−6 (1)	−3·7 (8)
C(6)—C(7)—C(8)—C(9)	78 (1)	−70·8 (7)
C(7)—C(8)—C(9)—N(1)	−86 (1)	90·0 (6)
C(8)—C(9)—N(1)—C(4)	55 (1)	−59·5 (6)
Ring *L* [atoms N(1), C(4), C(5), C(11), C(10), C(8) and C(9)]		
N(1)—C(4)—C(5)—C(11)	−33 (1)	35·6 (7)
C(4)—C(5)—C(11)—C(10)	76 (1)	−71·9 (7)
C(5)—C(11)—C(10)—C(8)	−4 (1)	−3·0 (7)
C(11)—C(10)—C(8)—C(9)	−74 (1)	76·6 (6)
C(10)—C(8)—C(9)—N(1)	40 (1)	−35·3 (6)
C(8)—C(9)—N(1)—C(4)	55 (1)	−59·5 (6)
C(9)—N(1)—C(4)—C(5)	−59 (1)	58·4 (6)
Ring *R* [atoms C(5), C(6), C(7), C(8), C(10) and C(11)]		
C(5)—C(6)—C(7)—C(8)	−6 (1)	−3·7 (8)
C(6)—C(7)—C(8)—C(10)	−48 (1)	55·5 (7)
C(7)—C(8)—C(10)—C(11)	52 (1)	−51·5 (6)
C(8)—C(10)—C(11)—C(5)	−4 (1)	−3·0 (7)
C(10)—C(11)—C(5)—C(6)	−48 (1)	54·3 (7)
C(11)—C(5)—C(6)—C(7)	54 (1)	−50·5 (7)

Energetically favoured configurations for cyclohexane are boat, chair and twisted boat; for cycloheptane chair, boat, twisted chair and twisted boat (Hendrickson, 1964). The azabicyclo[3.2.2]nonane ring adopts a boat–boat conformation, with both *L* and *R* rings adopting boat conformations while the base cycloheptane ring *B* adopts the chair conformation. The pyridine rings are planar.

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Structure of Morpholinium 5-Acetyl-3-cyano-1,4-dihydro-6-methyl-4-(2-nitrophenyl)-2-pyridinethiolate, $C_4H_{10}NO^+.C_{15}H_{12}N_3O_3S^-$

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Abstract. $M_r = 402{\cdot}46$, monoclinic, $C2/c$, $a = 21{\cdot}221\,(6)$, $b = 11{\cdot}665\,(4)$, $c = 16{\cdot}598\,(4)$ Å, $\beta = 108{\cdot}75\,(2)°$, $V = 3891\,(2)$ Å^3, $Z = 8$, $D_x = 1{\cdot}374$, $D_m = 1{\cdot}270$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 2{\cdot}027$ cm^{-1}, $F(000) = 1696$, $T = 293$ K. $R = 0{\cdot}052$ using 2913 independent observed reflections. The crystal has an ionic structure. Two conjugated systems N≡C—C=C—S and O=C—C=C—N exist in the anion and the heterocycle present in it has a flattened boat conformation. The length of the C(1)—S bond to the formally negatively charged S atom is equal to 1·725 (3) Å. The pseudo-axial *o*-nitrophenyl substituent is rotated by 75·6 (1)° with respect to the bottom of the boat. Intermolecular N—H···O, N—H···S and N—H···N hydrogen bonds of 2·729 (4), 3·407 (3) and 2·990 (4) Å are observed in the crystal.

Introduction. The title compound (I) was obtained by reaction of cyano(2-nitrobenzylidene)thioacetamide with acetylacetone in the presence of an excess of morpholine. Salt (I) is also formed on interaction of the enamine, obtained from acetylacetone and morpholine, with cyano(2-nitrobenzylidene)thioacetamide. The present X-ray study was performed to confirm the structure of salt (I).

Experimental. On recrystallization of salt (I) from ethanol prismatic ruby crystals were obtained, one of which with dimensions 0·2 × 0·2 × 0·3 mm was used to measure the cell parameters (using 24 reflections with $33 < 2\theta < 34°$) and intensities of 5381 reflections ($0 \le h \le 28$, $0 \le k \le 15$, $-22 \le l \le 22$) with a Syntex $P2_1$ diffractometer (Mo $K\alpha$, graphite monochromator, $\theta/2\theta$ scan, $2\theta_{\max} = 58°$). No significant variation in intensities of three standard reflections ($0\bar{2}0$, 202, 222) measured after every 100 reflections. No absorption and secondary-extinction corrections. Structure solved by direct methods (*MULTAN*, Germain, Main & Woolfson, 1971), revealing all nonhydrogen atoms, and refined by full-matrix least squares with anisotropic thermal parameters for nonhydrogen atoms, using 2913 independent reflections with $I > 2\sigma$ and minimizing $\sum w(|F_o|-|F_c|)^2$, $w = 1/[\sigma^2(F_o) + |F_o|^2]$. Scattering factors from *International Tables for X-ray Crystallography* (1974). Hydrogen atoms located by a difference synthesis and refined isotropically. Final *R*

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