Les huit atomes d'U de la maille se répartissent en deux groupes de position, U(1) et U(2). L'environnement de ces deux atomes est identique. Les distances et angles sont comparables, mais les facteurs d'agitation thermique des atomes de la molécule autour de U(2) sont légèrement plus élevés que ceux de la molécule autour de U(1). La Fig. 1 représente l'unité moléculaire autour de U(1): l'uranium est au centre d'une bipyramide hexagonale d'atomes d'oxygène dont les sommets sont les deux oxygènes de l'uranyle. Quatre oxygènes, O(11), O(12), O(11′) et O(12′), appartiennent aux deux nitrates bidentés et opposés, les deux autres, O(10) et O(10′), aux deux molécules d'amide monodenté. L'hexagone est plan mais non régulier: la distance entre les deux oxygènes du nitrate [O(11)–O(12): 2,14 Å] est plus courte que les distances O_{NO_3}–O_{amide} [O(10)–O(11): 2,57; O(10)–O(12′): 2,64 Å]. L'uranyle, linéaire, est perpendiculaire à l'hexagone. Les plans des ions nitrate sont pratiquement confondus avec celui de l'hexagone. L'oxygène du ligand amide est plus proche (2,35 Å) de l'uranium que les oxygènes du nitrate (2,50 Å), comme il a été observé dans les composés de ce type (Charpin *et al.*, 1985, 1986; Martin-Gil *et al.*, 1983). Les chaînes *n*-butyle ont une conformation *trans*: les moyennes des distances C—C et des angles C—C—C valent 1,48 (4) Å et 106 (3)° pour la molécule 1 et 1,41 (6) Å et 108 (4)° pour la molécule 2.

La Fig. 2 montre la projection de la structure suivant la direction [010]. Les deux sous réseaux d'atomes d'uranium constituent des plans mixtes d'U(1) et U(2). Dans le plan à la cote $z = 0$, U(2) se déduit de U(1) par une translation *xy* de $(\frac{1}{4}, -\frac{1}{4})$ alors que dans le plan à la cote $z = \frac{1}{2}$, la même opération s'effectue selon *xy* de $(\frac{1}{4}, \frac{1}{4})$, soit alternativement selon les deux diagonales du plan *xy*.

Nous remercions M. G. Gasparini (CEN Casaccia) qui nous a aimablement fourni un échantillon de *N*,*N*-di-*n*-butyl diméthyl-3,3 butanamide.

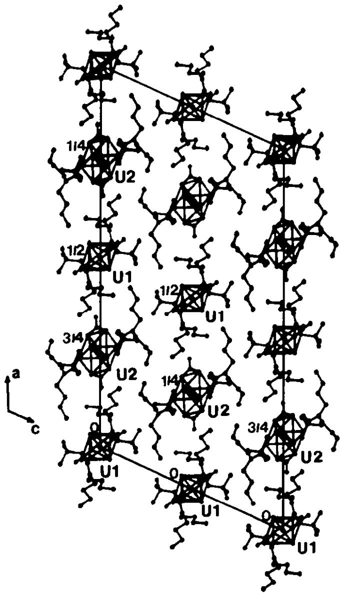

Fig. 2. Projection de la structure suivant la direction [010].

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Amminebis(pentamethylcyclopentadienyl)(thiophenolato)ytterbium(III)

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Abstract. $[Yb(C_{10}H_{15})_2(C_6H_5S)(NH_3)]$, $M_r = 569{\cdot}70$, triclinic, $P\bar{1}$, $a = 14{\cdot}091\ (6)$, $b = 18{\cdot}294\ (8)$, $c = 10{\cdot}430\ (6)$ Å, $\alpha = 104{\cdot}52\ (6)$, $\beta = 96{\cdot}66\ (5)$, $\gamma = 93{\cdot}77\ (5)°$, $V = 2572{\cdot}6\ (24)$ Å^3, $Z = 4$, $D_x = 1{\cdot}471$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 37{\cdot}0$ cm^{-1}, $F(000) = 1148$, $T = 296$ K, $R = 0{\cdot}038$ for 6052 unique

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reflections with $F^2 > 2\sigma(F^2)$. Two independent but chemically similar molecules are found in the asymmetric unit. The Yb is at the center of a distorted tetrahedron consisting of an S atom, an N atom and the centers of two pentamethylcyclopentadienyl rings. Distances are: Yb—N 2·423 (8) and 2·432 (8) Å; Yb—S 2·670 (3) and 2·679 (3) Å; average Yb—C 2·64 ±0·02 Å; average Yb—Cp 2·347 ±0·007 Å.

Introduction. The title compound was prepared as part of an extensive study of the electron-transfer and bond-cleaving properties of the divalent ytterbium metallocene, $[(CH_3)_5C_5]_2Yb$, towards transition-metal carbonyls with metal to metal bonds (Boncella & Andersen, 1984) and with organic compounds with non-metal to non-metal bonds (Berg, 1986).

Experimental. The trivalent Yb complex was prepared from the reaction of $[(CH_3)_5C_5]_2Yb(NH_3)_2$ and $C_6H_5SSC_6H_5$ in 2:1 molar ratio in diethyl ether, and red crystals were obtained by crystallization from hexane (263 K). The synthesis details and spectroscopic characterization of this and related molecules will be published in due course (Berg, 1986). The red, air-sensitive crystals used in the X-ray study were picked from this batch and sealed inside quartz capillaries under argon. Crystal 0·09 × 0·19 × 0·36 mm with six faces; modified Picker automatic diffractometer, graphite monochromator; cell dimensions from 27 reflections, $23° < 2\theta < 34°$; analytical absorption correction, range 1·33 to 2·10; max. $(\sin\theta)/\lambda = 0{\cdot}60$ Å^{-1}, h −16 to 16, k −21 to 21, l −12 to 7; three standard reflections, no decay, 4% random variation in intensities from average; 15 867 data, 9132 unique, $R_{\rm int} = 0{\cdot}031$; structure solved by Patterson and Fourier methods; refined on F using 6052 reflections for which $F^2 > 2\sigma(F^2)$, 523 parameters; 16 non-methyl H atoms in calculated positions with fixed isotropic thermal parameters, methyl H atoms were not observed in difference Fourier map and their positions could not be unambiguously estimated; anisotropic thermal parameters for non-hydrogen atoms; $R = 0{\cdot}075$ (all data), $R = 0{\cdot}038$ [$F^2 > 2\sigma(F^2)$ data], $wR = 0{\cdot}048$, $S = 1{\cdot}2$; $w = 4F_o^2/[\sigma^2(F_o^2) + (0{\cdot}06F_o^2)^2]$; max. $(\text{shift}/\sigma) = 0{\cdot}002$; empirical extinction correction, $F_{\rm corr} = (1 + 8{\cdot}4 \times 10^{-8}I)F_o$; max. and min. of ΔF synthesis 0·6 and −0·7 e Å^{-3}; atomic f for neutral Yb, S, N and C, and spherical bonded H from *International Tables for X-ray Crystallography* (1974); local unpublished programs and *ORTEP* (Johnson, 1965). Atomic parameters are listed in Table 1,* and distances and angles are listed in Table 2. Figs. 1 and 2 show the molecules and the numbering scheme.

Discussion. The asymmetric unit contains two crystallographically independent molecules that are chemically the same. The most obvious geometrical differences can be seen in Figs. 1 and 2 in which the two molecules are in a similar orientation. The disposition of the thiophenolato groups are such that reflection of the group through a mirror in the S—Yb—N plane in one of the molecules would bring it into near geometrical equivalence with the other molecule. Comparable distances in the two molecules are statistically equivalent with no differences exceeding 3 e.s.d.'s. Most of the corresponding angles are similar, with the exception of the

Table 1. *Atomic parameters*

$$B_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
Yb(1)	0·19533 (2)	0·63154 (2)	0·21092 (3)	3·40 (1)
S(1)	0·16339 (19)	0·52104 (15)	−0·01607 (24)	5·55 (8)
N(1)	0·0234 (5)	0·6283 (5)	0·1511 (8)	5·4 (3)
C(1)	0·2844 (7)	0·7695 (5)	0·2915 (9)	4·8 (3)
C(2)	0·3347 (6)	0·7300 (5)	0·1888 (10)	4·6 (3)
C(3)	0·2730 (7)	0·7142 (5)	0·0668 (9)	4·7 (3)
C(4)	0·1844 (7)	0·7421 (5)	0·0926 (9)	4·5 (3)
C(5)	0·1901 (7)	0·7762 (4)	0·2319 (10)	4·8 (3)
C(6)	0·3324 (9)	0·8137 (6)	0·4321 (10)	6·9 (4)
C(7)	0·4411 (7)	0·7181 (6)	0·2021 (13)	7·0 (4)
C(8)	0·3018 (10)	0·6841 (6)	−0·0737 (11)	7·7 (4)
C(9)	0·1016 (8)	0·7418 (7)	−0·0161 (11)	7·2 (4)
C(10)	0·1120 (9)	0·8142 (7)	0·3055 (13)	7·5 (4)
C(11)	0·2897 (7)	0·6030 (6)	0·4250 (8)	4·7 (3)
C(12)	0·2572 (7)	0·5315 (5)	0·3334 (9)	4·4 (3)
C(13)	0·1557 (7)	0·5191 (5)	0·3205 (9)	4·6 (3)
C(14)	0·1252 (7)	0·5861 (5)	0·4054 (9)	4·9 (3)
C(15)	0·2075 (8)	0·6362 (6)	0·4678 (8)	5·1 (3)
C(16)	0·3927 (8)	0·6313 (7)	0·4850 (12)	7·0 (4)
C(17)	0·3217 (8)	0·4715 (6)	0·2710 (11)	6·4 (4)
C(18)	0·0928 (8)	0·4479 (6)	0·2423 (10)	6·1 (3)
C(19)	0·0213 (9)	0·5968 (8)	0·4378 (12)	7·7 (5)
C(20)	0·2035 (10)	0·7069 (7)	0·5879 (11)	7·6 (4)
C(21)	0·2477 (7)	0·5024 (5)	−0·1287 (9)	4·4 (3)
C(22)	0·2215 (7)	0·5015 (6)	−0·2593 (10)	5·4 (3)
C(23)	0·2854 (11)	0·4831 (7)	−0·3516 (11)	7·3 (4)
C(24)	0·3756 (12)	0·4673 (9)	−0·3098 (15)	8·9 (6)
C(25)	0·4020 (10)	0·4690 (9)	−0·1815 (16)	9·0 (6)
C(26)	0·3395 (9)	0·4857 (7)	−0·0906 (11)	7·0 (4)
Yb(2)	0·23925 (2)	0·08118 (2)	0·16928 (3)	3·39 (1)
S(2)	0·10569 (17)	0·09900 (16)	−0·02235 (25)	5·29 (8)
N(2)	0·1425 (5)	−0·0406 (4)	0·1085 (7)	4·9 (2)
C(27)	0·2801 (7)	0·1501 (7)	0·4262 (8)	5·4 (3)
C(28)	0·2302 (10)	0·2017 (5)	0·3692 (10)	6·1 (4)
C(29)	0·1333 (7)	0·1698 (6)	0·3259 (8)	5·0 (3)
C(30)	0·1233 (7)	0·0986 (6)	0·3556 (9)	5·3 (3)
C(31)	0·2145 (8)	0·0858 (6)	0·4176 (8)	5·2 (3)
C(32)	0·3751 (10)	0·1735 (11)	0·5172 (13)	11·3 (6)
C(33)	0·2679 (12)	0·2833 (6)	0·3738 (13)	9·8 (6)
C(34)	0·0467 (12)	0·2077 (10)	0·2800 (13)	12·2 (7)
C(35)	0·0298 (9)	0·0480 (9)	0·3449 (13)	9·7 (5)
C(36)	0·2385 (12)	0·0206 (7)	0·4799 (12)	9·4 (6)
C(37)	0·4213 (6)	0·1071 (6)	0·1484 (12)	6·0 (4)
C(38)	0·4150 (6)	0·0384 (6)	0·1869 (10)	5·3 (3)
C(39)	0·3615 (7)	−0·0170 (5)	0·0818 (12)	5·5 (3)
C(40)	0·3347 (7)	0·0164 (7)	−0·0242 (10)	5·8 (3)
C(41)	0·3714 (7)	0·0930 (7)	0·0152 (11)	5·6 (4)
C(42)	0·4862 (9)	0·1803 (8)	0·2222 (17)	11·1 (6)
C(43)	0·4716 (9)	0·0219 (10)	0·3072 (13)	10·0 (6)
C(44)	0·3472 (11)	−0·1020 (7)	0·0732 (20)	11·4 (7)
C(45)	0·2831 (10)	−0·0290 (11)	−0·1633 (13)	11·7 (7)
C(46)	0·3727 (10)	0·1478 (10)	−0·0734 (17)	10·8 (7)
C(47)	0·1399 (6)	0·1724 (5)	−0·0979 (8)	4·3 (3)
C(48)	0·1734 (8)	0·2441 (6)	−0·0244 (9)	5·7 (3)
C(49)	0·1990 (9)	0·2993 (6)	−0·0860 (12)	6·8 (4)
C(50)	0·1906 (8)	0·2850 (7)	−0·2199 (12)	6·4 (4)
C(51)	0·1558 (8)	0·2140 (7)	−0·2950 (10)	6·2 (4)
C(52)	0·1299 (7)	0·1582 (6)	−0·2359 (9)	5·1 (3)

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* Lists of structure factors, anisotropic thermal parameters, calculated hydrogen positions, distances and angles, least-squares planes and additional *ORTEP* drawings have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43375 (49 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected distances* (Å) *and angles* (°)

Yb(1)—N(1)	2·423 (8)	Yb(2)—N(2)	2·432 (8)
Yb(1)—S(1)	2·670 (3)	Yb(2)—S(2)	2·679 (3)
Yb(1)—C(1)	2·632 (8)	Yb(2)—C(27)	2·637 (8)
Yb(1)—C(2)	2·646 (7)	Yb(2)—C(28)	2·650 (8)
Yb(1)—C(3)	2·654 (8)	Yb(2)—C(29)	2·661 (8)
Yb(1)—C(4)	2·624 (8)	Yb(2)—C(30)	2·654 (8)
Yb(1)—C(5)	2·608 (8)	Yb(2)—C(31)	2·635 (8)
Yb(1)—C(11)	2·649 (8)	Yb(2)—C(37)	2·621 (8)
Yb(1)—C(12)	2·620 (8)	Yb(2)—C(38)	2·646 (8)
Yb(1)—C(13)	2·651 (8)	Yb(2)—C(39)	2·641 (8)
Yb(1)—C(14)	2·643 (8)	Yb(2)—C(40)	2·632 (8)
Yb(1)—C(15)	2·644 (8)	Yb(2)—C(41)	2·629 (8)
S(1)—C(21)	1·756 (9)	S(2)—C(47)	1·786 (9)
Yb(1)—Cp_1^*	2·340	Yb(2)—Cp_3	2·355
Yb(1)—Cp_2	2·349	Yb(2)—Cp_4	2·343
S(1)—Yb(1)—N(1)	78·3 (2)	S(2)—Yb(2)—N(2)	78·1 (2)
Cp_1—Yb(1)—Cp_2	136·2	Cp_3—Yb(2)—Cp_4	136·9
Cp_1—Yb(1)—S(1)	111·4	Cp_3—Yb(2)—S(2)	108·6
Cp_1—Yb(1)—N(1)	102·8	Cp_3—Yb(2)—N(2)	103·1
Cp_2—Yb(1)—S(1)	108·1	Cp_4—Yb(2)—S(2)	110·7
Cp_2—Yb(1)—N(1)	102·9	Cp_4—Yb(2)—N(2)	101·5
Yb(1)—S(1)—C(21)	123·0 (3)	Yb(2)—S(2)—C(47)	113·9 (3)

* Cp_1, Cp_2, Cp_3 and Cp_4 represent the centroids of cyclopentadienyl atoms C(1)–C(5), C(11)–C(15), C(27)–C(31) and C(27)–C(31), respectively.

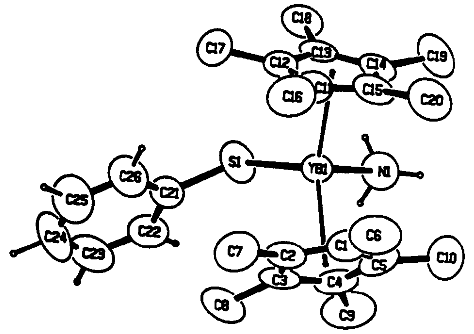

Fig. 1. *ORTEP* (Johnson, 1965) drawing of molecule I showing the atomic numbering scheme; 50% probability ellipsoids.

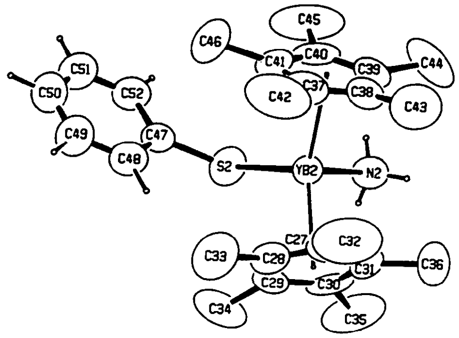

Fig. 2. *ORTEP* (Johnson, 1965) drawing of molecule II showing the atomic numbering scheme; 50% probability ellipsoids.

Yb—S—C angles that differ by 9°, which is probably due to steric effects in the different environments of the thiophenolato groups. The bond lengths in the C_6H_5S portion of the molecule are essentially unchanged from those in $C_6H_5SSC_6H_5$ (Sacerdoti & Gilli, 1975), $C_6H_5SCH_3$ (Zaripov, 1976) and C_6H_5SCl (Zaripov, Popik & Vilkov, 1980). The Yb—S—C bond angles in the two individual molecules in the unit cell are opened *ca* 15° relative to the angle at sulfur in $C_6H_5SSC_6H_5$, $C_6H_5SCH_3$ and C_6H_5SCl.

The cyclopentadienyl and phenyl rings are planar. The cyclopentadienyl rings that sandwich the Yb atom are staggered. The methyl C atoms are bent out of the planes of the cyclopentadienyl rings in a direction away from the Yb atom. The displacement of these C atoms from the least-squares planes of the cyclopentadienyl rings range from 0·0 to 0·4 Å with an average displacement for all 20 methyl C atoms of 0·19 ±0·09 Å. This outward bending of the methyl groups was also observed in $[(CH_3)_5C_5]_2Yb(OC_4H_8)$ (Tilley, Andersen, Spencer, Ruben, Zalkin & Templeton, 1980) and in $[(CH_3)_5C_5]_2Yb(NC_5H_5)_2$ (Tilley, Andersen, Spencer & Zalkin, 1982) in which the displacement from the cyclopentadienyl planes was about half the amount reported here.

The averaged Yb—S and Yb—C distances in the two independent molecules of $[(CH_3)_5C_5]_2Yb[S(C_6H_5)](NH_3)$ of 2·675 ±0·005 Å and 2·64 ±0·02 Å are similar to those found in $[(CH_3)_5C_5]_2YbS_2CN(C_2H_5)_2$ of 2·70 (1) Å and 2·63 ±0·03 Å, respectively (Tilley, Andersen, Zalkin & Templeton, 1982). The Yb—S distance is shorter than the Lu—S distance of 2·715 ±0·007 Å found in $Li(OC_4H_8)_2[(CH_3)_5C_5]_2Lu[SC(CH_3)_3]_2$ (Schumann, Albrecht & Hahn, 1985) as expected since Lu^{III} is *ca* 0·01 Å smaller than Yb^{III} (Shannon, 1976). The averaged Yb—N distance of 2·428 ±0·003 Å is shorter than the analogous distance of 2·57 ±0·01 Å in $[(CH_3)_5C_5]_2Yb(NC_5H_5)_2$ (Tilley, Andersen, Spencer & Zalkin, 1982) and 2·55 (3) Å in $[(CH_3)_5C_5]_2Yb(NH_3)(OC_4H_8)$ (Wanda, Dye & Rogers, 1984). This trend is expected since the radius of Yb^{III} is *ca* 0·15 Å smaller than that of Yb^{II} for a given coordination number (Shannon, 1976).

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Structure of Bis(2-methyl-1,2-propanediamine)nickel(II) Diperchlorate

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Abstract. [$Ni(C_4H_{12}N_2)_2$](ClO_4)$_2$, M_r = 433·92, monoclinic, space group $P2_1/c$, a = 14·0377 (21), b = 13·0379 (10), c = 20·2665 (17) Å, β = 108·11 (1)°, V = 3525·4 (7) Å^3, Z = 8, D_m = 1·65 (1), D_x = 1·63 Mg m^{-3}, Cu $K\alpha$ radiation (graphite crystal monochromator, λ = 1·54184 Å), μ = 4·837 mm^{-1}, T = 290 K, F(000) = 1808. Final R = 0·072, wR = 0·079 for 3724 'observed' reflections and 522 variables. The structure consists of two [$Ni(C_4H_{12}N_2)_2$]$^{2+}$ cations and four (ClO_4)$^-$ anions in the asymmetric unit. The central Ni atom is square-planar coordinated, by two 2-methyl-1,2-propanediamine ligands. Distances and angles are normal [averages Ni—N(CMe_2) 1·905 (8) Å, Ni—N(CH_2) 1·916 (9) Å and N—Ni—N bite angle, 86·0 (4)°]. The perchlorate anions are disordered; also two terminal methyl groups have abnormally large temperature factors.

Introduction. As a continuation of our investigation on the structures of Ni complexes, the crystal structure of a complex with 2-methyl-1,2-propanediamine has been determined. In previous work (García-Granda & Gómez-Beltrán, 1984) we have determined the crystal structure of bis(2-methyl-1,2-propanediamine-*N,N'*)-bis(trichloroacetato-*O*)nickel(II), an octahedral complex with the same diamine as ligand. This investigation was undertaken in order to obtain enough structural data to understand the chemical and physicochemical behaviour of Ni in complexes with different C-substituted derivatives of ethylenediamine.

Experimental. A yellow crystal of approximately 0·25 × 0·20 × 0·08 mm was used for the measurements. Density measured by flotation. Throughout the experiment Cu $K\alpha$ radiation was used with a graphite crystal monochromator on a Nonius CAD-4 single-crystal diffractometer (λ = 1·54184 Å). The unit-cell dimensions were determined from the angular settings of 25 reflections. The intensities of 13 652 reflections (one half sphere up to θ = 70°), hkl range from (−17,−15,0) to (17,15,24) were measured, using the ω–2θ scan technique, with a scan angle of 1·50° and a variable scan rate with a maximum scan time of 60 s per reflection. The intensity of the primary beam was checked throughout the data collection by monitoring three standard reflections every 30 min. The final drift correction factors were between 1·00 and 1·24. On all reflections profile analysis was performed (Lehmann & Larsen, 1974; Grant & Gabe, 1978); empirical absorption correction was applied, using ψ scans (North, Phillips & Mathews, 1968), μ(Cu $K\alpha$) = 4·837 mm^{-1} (correction factors were in the range 1·00 to 0·55). Symmetry-equivalent reflections were averaged, $R_{\rm int} = \sum(I - \langle I \rangle)/\sum I$ = 0·039, resulting in 6665 unique reflections of which 3724 were observed with $I > 3\sigma(I)$. Lorentz and polarization corrections were applied and the data were reduced to $|F_o|$ values.

The structure was solved automatically, using *PATSYS* (Behm & Beurskens, 1985): the positions of the two Ni atoms were located from the Patterson map (Sheldrick, 1983), and input to *DIRDIF* (Beurskens *et al.*, 1982), and the positions of all non-hydrogen atoms were obtained. From the beginning of the refinement exceptionally high temperature factors for the O atoms

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