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O-Pivaloyl diphenylselenophosphinate

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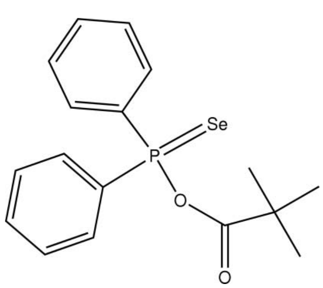
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.006 Å; R factor = 0.057; wR factor = 0.147; data-to-parameter ratio = 19.0.

The title compound, $C_{17}H_{19}O_2PSe$, was obtained in the reaction of the diphenylmonoselenophosphinic acid ammonium salt with pivaloyl chloride. The P—Se bond length of 2.0769 (11) Å is normal, while the P—O bond length of 1.650 (3) Å is longer than in related *O*-alkyl and *O*-aryl derivatives. One phenyl ring is periplanar to the Se—P—C plane, while the dihedral angle between the two phenyl rings is *ca* 73°. The carbonyl group is in a synperiplanar position [torsion angle = 8.9 (6)°] to one of the methyl groups of the pivaloyl group. This is the first *O*-acyl derivative of diphenylmonoselenophosphinic acid characterized by X-ray structural analysis.

Related literature

Syntheses and the chemical properties of *O*-acyl monoselenophosphates have already been described by Rachon *et al.* (2005). For other monoselenophosphates, such as *O*-alkyl or *O*-aryl esters, see: Lepicard *et al.* (1969); Balakrishna *et al.* (2002, 2005); Mague *et al.* (2007). For details of the Cambridge Crystallographic Database, see: Allen (2002).



Experimental

Crystal data

$C_{17}H_{19}O_2PSe$
M_r = 365.25
Monoclinic, $P2_1/c$
a = 9.6212 (5) Å
b = 10.3914 (5) Å
c = 17.1087 (9) Å
β = 99.618 (5)°
V = 1686.45 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.32 mm^{-1}
T = 120 K
0.22 × 0.2 × 0.12 mm

Data collection

Oxford Diffraction KM-4-CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)
T_{min} = 0.588, T_{max} = 0.760
12450 measured reflections
3674 independent reflections
2596 reflections with $I > 2\sigma(I)$
R_{int} = 0.06

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.147
S = 0.97
3674 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

Table 1
Comparison of the geometry of the title compound with related compounds (Å, °).

CSD refcode (Allen, 2002)	P—Se	P—O	Ph–Ph dihedral	Smaller torsion	Reference
	2.0769 (11)	1.650 (3)	72.64 (14)	7.0 (4)	This work
MPSEPO	2.080	1.619	82.62	4.15	Lepicard *et al.* (1969)
MUMFUV	2.072	1.624	80.93	13.32	Balakrishna *et al.* (2002)
	2.070	1.612	75.01	22.34	
RAMXEJ	2.089	1.596	78.65	8.84	Balakrishna *et al.* (2005)
	2.079	1.585	78.49	6.58	
YIQOM	2.089	1.620	70.15	6.15	Mague *et al.* (2007)

Data collection: *CrysAlis CCD* (Oxford Diffraction 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2163).

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Acta Cryst. (2009). E**65**, o853–o854 [doi:10.1107/S1600536809009295]

O-Pivaloyl diphenylselenophosphinate

Grzegorz Cholewinski, Jaroslaw Chojnacki, Jerzy Pikies and Janusz Rachon

S1. Comment

O-acyl monoselenophosphates were studied in a search for potential selenoacylating agents. *O*-pivaloyl-diphenylmono-selenophosphinate, $C_{17}H_{19}O_2PSe$, was obtained in the reaction of diphenylmonoselenophosphinic acid ammonium salt with pivaloyl chloride (Rachon *et al.*, 2005). The P—Se bond length is normal for a double bond, while the P—O bond is rather long when compared with the related *O*-alkyl and *O*-aryl derivatives (see Table 1). One phenyl ring is placed periplanar to the Se—P—C plane, while the dihedral angle between the two phenyl rings is relatively small. The carbonyl group is in a synperiplanar position [torsion angle = 8.9 (6)°] to one of methyl groups in the pivaloyl group.

This compound, together with *O*-*p*-chlorobenzoyl-diphenylselenophosphinate, reported in the following paper, are the first structures determined by X-ray diffraction of *O*-acyl derivatives of diphenylmonoselenophosphinic acid reported. Only four related *O*-alkyl and *O*-aryl derivatives were characterized by *x*-ray study so far: methyl diphenylseleno-phosphinate (Lepicard *et al.*, 1969), 1,4-bis(diphenyl(seleno)phosphinito)cyclohexane (Balakrishna *et al.*, 2005); 1,1′-methylene-bis(2-((diphenylphosphoroselenoyl)oxy)naphthalene) (Balakrishna *et al.*, 2002) and *O*-2-naphthyl diphenyl-selenophosphinate (Mague *et al.*, 2007).

S2. Experimental

O-Pivaloyl diphenylmonoselenophosphinate was obtained in the reaction of diphenylmonoselenophosphinic acid ammonium salt with pivaloyl chloride with 43% yield (Rachon *et al.*, 2005, compound numbered as 2u, melting point 63-65 °C). Relevant ^{1}H, ^{13}C, ^{31}P NMR, MS and IR spectra were recorded and are consistent with the formula anticipated - see the supporting information for the article cited.

S3. Refinement

Hydrogen atoms were placed in calculated positions and refined using a standard riding model. C–H bond lengths were set to 0.98 or 0.95 Å and U_{iso}(H) were set to 1.5 or 1.2 U_{eq}(C) for methyl or aromatic C–H groups, respectively.

The residual electron density peak is 0.84 Å from Se1, the deepest electron density hole is 1.24 Å from Se1.

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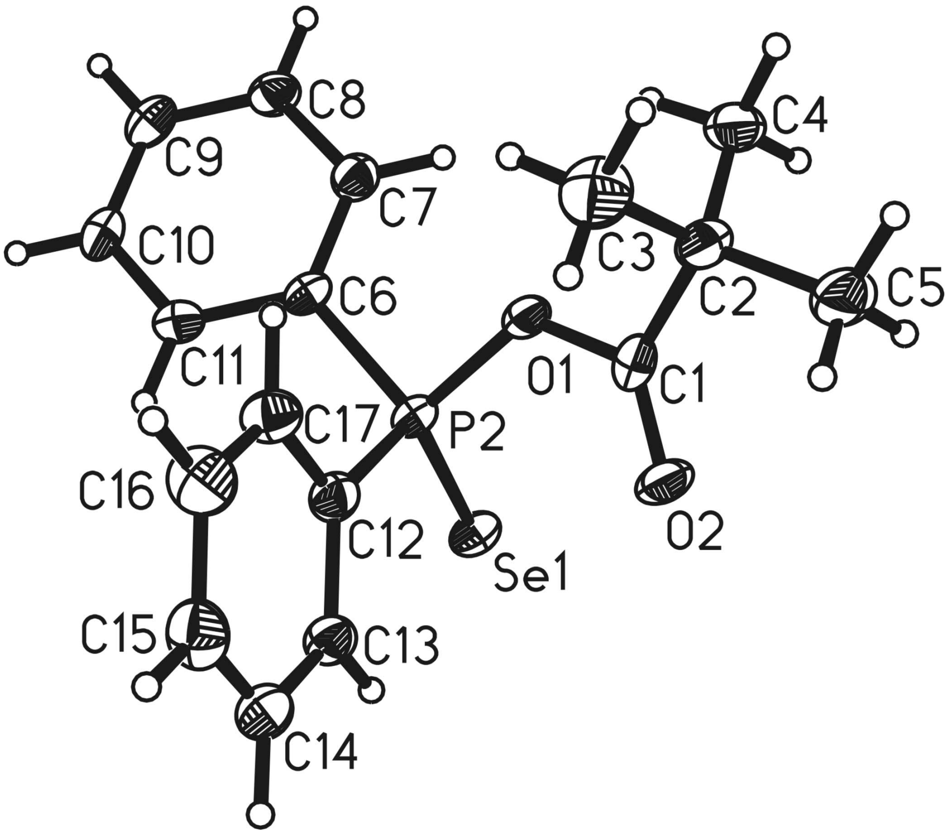

Figure 1

View of the title compound showing the atom-numbering scheme (50% probability displacement ellipsoids).

O-pivaloyl diphenylselenophosphinate

Crystal data

$C_{17}H_{19}O_2PSe$	$F(000) = 744$
$M_r = 365.25$	$D_x = 1.439$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 337(2) K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.6212$ (5) Å	Cell parameters from 5361 reflections
$b = 10.3914$ (5) Å	$\theta = 2.0$–$32.4°$
$c = 17.1087$ (9) Å	$\mu = 2.32$ mm^{-1}
$\beta = 99.618$ (5)°	$T = 120$ K
$V = 1686.45$ (15) Å^3	Fragment, colourless
$Z = 4$	0.22 × 0.2 × 0.12 mm

Data collection

Oxford Diffraction KM-4-CCD diffractometer	12450 measured reflections
Radiation source: Mo Kα radiation	3674 independent reflections
Graphite monochromator	2596 reflections with $I > 2\sigma(I)$
ω scans (0.75° width)	$R_{int} = 0.06$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)	$\theta_{max} = 27°$, $\theta_{min} = 2.3°$
$T_{min} = 0.588$, $T_{max} = 0.760$	$h = -12 \rightarrow 12$
	$k = -12 \rightarrow 13$
	$l = -21 \rightarrow 19$

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Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.147$
$S = 0.97$
3674 reflections
193 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0979P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 2.18$ e Å^{-3}
$\Delta\rho_{min} = -0.65$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
P2	0.85844 (10)	0.75002 (9)	0.28847 (6)	0.0208 (2)
Se1	0.78275 (4)	0.76292 (4)	0.16772 (2)	0.02698 (17)
O1	0.8422 (2)	0.6100 (2)	0.33157 (15)	0.0243 (6)
O2	0.6042 (3)	0.6037 (3)	0.30807 (16)	0.0310 (6)
C1	0.7130 (4)	0.5607 (4)	0.3426 (2)	0.0243 (8)
C2	0.7308 (4)	0.4557 (4)	0.4043 (2)	0.0274 (8)
C3	0.7855 (5)	0.5221 (5)	0.4839 (2)	0.0424 (11)
H3A	0.8789	0.5588	0.4826	0.064*
H3B	0.7923	0.4588	0.5269	0.064*
H3C	0.7203	0.5908	0.493	0.064*
C4	0.8353 (4)	0.3542 (4)	0.3869 (3)	0.0341 (10)
H4A	0.7989	0.3118	0.3365	0.051*
H4B	0.8486	0.2903	0.4296	0.051*
H4C	0.9258	0.3953	0.3835	0.051*
C5	0.5874 (4)	0.3949 (4)	0.4078 (3)	0.0412 (11)
H5A	0.5226	0.4609	0.4212	0.062*
H5B	0.5984	0.3272	0.4482	0.062*
H5C	0.5493	0.3574	0.356	0.062*
C6	1.0474 (4)	0.7623 (3)	0.3126 (2)	0.0211 (8)
C7	1.1309 (4)	0.6520 (4)	0.3187 (2)	0.0263 (8)
H7	1.0889	0.569	0.3136	0.032*
C8	1.2769 (4)	0.6654 (4)	0.3324 (2)	0.0273 (9)
H8	1.3348	0.5908	0.3365	0.033*
C9	1.3386 (4)	0.7856 (4)	0.3399 (2)	0.0298 (9)
H9	1.4384	0.7935	0.3494	0.036*
C10	1.2550 (4)	0.8946 (4)	0.3337 (2)	0.0277 (9)
H10	1.2976	0.9773	0.3387	0.033*
C11	1.1095 (4)	0.8839 (4)	0.3203 (2)	0.0242 (8)

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H11	1.0524	0.9589	0.3164	0.029*
C12	0.7863 (4)	0.8624 (4)	0.3506 (2)	0.0254 (8)
C13	0.6748 (4)	0.9438 (4)	0.3208 (2)	0.0262 (8)
H13	0.6364	0.9409	0.266	0.031*
C14	0.6194 (4)	1.0279 (4)	0.3691 (3)	0.0338 (10)
H14	0.5447	1.084	0.3476	0.041*
C15	0.6734 (4)	1.0307 (4)	0.4499 (3)	0.0357 (10)
H15	0.6343	1.0875	0.4839	0.043*
C16	0.7843 (5)	0.9505 (4)	0.4806 (3)	0.0376 (10)
H16	0.8216	0.953	0.5356	0.045*
C17	0.8406 (4)	0.8673 (4)	0.4316 (2)	0.0307 (9)
H17	0.9169	0.8128	0.453	0.037*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P2	0.0152 (5)	0.0257 (5)	0.0208 (5)	0.0002 (4)	0.0014 (4)	0.0001 (4)
Se1	0.0227 (2)	0.0355 (3)	0.0210 (2)	0.00040 (17)	−0.00147 (16)	0.00093 (15)
O1	0.0153 (13)	0.0272 (13)	0.0289 (14)	−0.0003 (10)	−0.0002 (10)	0.0038 (11)
O2	0.0159 (14)	0.0380 (16)	0.0371 (16)	0.0016 (12)	−0.0016 (12)	0.0088 (13)
C1	0.0201 (19)	0.0271 (19)	0.026 (2)	−0.0059 (16)	0.0044 (16)	−0.0059 (15)
C2	0.0211 (19)	0.035 (2)	0.025 (2)	−0.0008 (17)	0.0007 (16)	0.0055 (16)
C3	0.050 (3)	0.050 (3)	0.025 (2)	−0.004 (2)	0.003 (2)	0.0038 (19)
C4	0.026 (2)	0.035 (2)	0.040 (2)	0.0042 (18)	0.0028 (18)	0.0097 (18)
C5	0.023 (2)	0.046 (3)	0.054 (3)	−0.0007 (19)	0.006 (2)	0.017 (2)
C6	0.0152 (17)	0.0296 (19)	0.0183 (18)	0.0002 (14)	0.0024 (14)	0.0004 (14)
C7	0.023 (2)	0.0282 (19)	0.027 (2)	−0.0011 (16)	0.0032 (16)	0.0007 (15)
C8	0.020 (2)	0.031 (2)	0.031 (2)	0.0051 (16)	0.0040 (16)	−0.0002 (16)
C9	0.019 (2)	0.041 (2)	0.028 (2)	−0.0034 (17)	0.0029 (16)	−0.0027 (18)
C10	0.023 (2)	0.033 (2)	0.025 (2)	−0.0076 (16)	0.0008 (16)	−0.0022 (16)
C11	0.022 (2)	0.0279 (19)	0.0225 (19)	0.0044 (16)	0.0031 (15)	0.0010 (15)
C12	0.020 (2)	0.0263 (19)	0.030 (2)	0.0003 (16)	0.0039 (16)	−0.0025 (16)
C13	0.0209 (19)	0.028 (2)	0.030 (2)	0.0002 (16)	0.0040 (16)	0.0016 (16)
C14	0.021 (2)	0.032 (2)	0.048 (3)	−0.0008 (17)	0.0052 (19)	−0.0006 (19)
C15	0.035 (2)	0.035 (2)	0.039 (3)	−0.0017 (19)	0.013 (2)	−0.0078 (19)
C16	0.047 (3)	0.039 (2)	0.028 (2)	0.002 (2)	0.0062 (19)	−0.0013 (18)
C17	0.030 (2)	0.034 (2)	0.026 (2)	0.0029 (18)	0.0008 (17)	0.0022 (17)

Geometric parameters (Å, º)

P2—O1	1.650 (3)	C7—C8	1.392 (5)
P2—C12	1.795 (4)	C7—H7	0.95
P2—C6	1.801 (4)	C8—C9	1.381 (6)
P2—Se1	2.0769 (11)	C8—H8	0.95
O1—C1	1.386 (4)	C9—C10	1.383 (6)
O2—C1	1.200 (4)	C9—H9	0.95
C1—C2	1.507 (5)	C10—C11	1.384 (5)
C2—C4	1.521 (5)	C10—H10	0.95

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C2—C5	1.527 (5)	C11—H11	0.95
C2—C3	1.539 (5)	C12—C13	1.394 (5)
C3—H3A	0.98	C12—C17	1.398 (5)
C3—H3B	0.98	C13—C14	1.370 (5)
C3—H3C	0.98	C13—H13	0.95
C4—H4A	0.98	C14—C15	1.394 (6)
C4—H4B	0.98	C14—H14	0.95
C4—H4C	0.98	C15—C16	1.386 (6)
C5—H5A	0.98	C15—H15	0.95
C5—H5B	0.98	C16—C17	1.377 (6)
C5—H5C	0.98	C16—H16	0.95
C6—C7	1.393 (5)	C17—H17	0.95
C6—C11	1.395 (5)		

O1—P2—C12	103.55 (16)	C7—C6—P2	120.4 (3)
O1—P2—C6	97.32 (15)	C11—C6—P2	119.1 (3)
C12—P2—C6	107.07 (17)	C8—C7—C6	118.9 (3)
O1—P2—Se1	117.24 (10)	C8—C7—H7	120.5
C12—P2—Se1	116.17 (13)	C6—C7—H7	120.5
C6—P2—Se1	113.35 (13)	C9—C8—C7	120.8 (4)
C1—O1—P2	122.8 (2)	C9—C8—H8	119.6
O2—C1—O1	121.6 (3)	C7—C8—H8	119.6
O2—C1—C2	127.0 (3)	C8—C9—C10	119.9 (4)
O1—C1—C2	111.3 (3)	C8—C9—H9	120
C1—C2—C4	111.5 (3)	C10—C9—H9	120
C1—C2—C5	109.3 (3)	C9—C10—C11	120.4 (4)
C4—C2—C5	110.6 (3)	C9—C10—H10	119.8
C1—C2—C3	106.1 (3)	C11—C10—H10	119.8
C4—C2—C3	110.1 (3)	C10—C11—C6	119.6 (3)
C5—C2—C3	109.2 (3)	C10—C11—H11	120.2
C2—C3—H3A	109.5	C6—C11—H11	120.2
C2—C3—H3B	109.5	C13—C12—C17	118.5 (4)
H3A—C3—H3B	109.5	C13—C12—P2	121.9 (3)
C2—C3—H3C	109.5	C17—C12—P2	119.5 (3)
H3A—C3—H3C	109.5	C14—C13—C12	121.4 (4)
H3B—C3—H3C	109.5	C14—C13—H13	119.3
C2—C4—H4A	109.5	C12—C13—H13	119.3
C2—C4—H4B	109.5	C13—C14—C15	119.5 (4)
H4A—C4—H4B	109.5	C13—C14—H14	120.2
C2—C4—H4C	109.5	C15—C14—H14	120.2
H4A—C4—H4C	109.5	C16—C15—C14	119.9 (4)
H4B—C4—H4C	109.5	C16—C15—H15	120
C2—C5—H5A	109.5	C14—C15—H15	120
C2—C5—H5B	109.5	C17—C16—C15	120.2 (4)
H5A—C5—H5B	109.5	C17—C16—H16	119.9
C2—C5—H5C	109.5	C15—C16—H16	119.9
H5A—C5—H5C	109.5	C16—C17—C12	120.4 (4)
H5B—C5—H5C	109.5	C16—C17—H17	119.8

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C7—C6—C11	120.4 (3)	C12—C17—H17	119.8
Se1—P2—O1—C1	−69.5 (3)	O2—C1—C2—C3	−108.5 (5)
C6—P2—O1—C1	169.4 (3)	O2—C1—C2—C4	131.6 (4)
C12—P2—O1—C1	59.8 (3)	O2—C1—C2—C5	8.9 (6)
Se1—P2—C6—C7	−92.1 (3)	P2—C6—C7—C8	175.9 (3)
Se1—P2—C6—C11	84.0 (3)	C11—C6—C7—C8	−0.1 (5)
O1—P2—C6—C7	31.8 (3)	P2—C6—C11—C10	−175.8 (3)
O1—P2—C6—C11	−152.1 (3)	C7—C6—C11—C10	0.3 (5)
C12—P2—C6—C7	138.5 (3)	C6—C7—C8—C9	0.0 (5)
C12—P2—C6—C11	−45.4 (3)	C7—C8—C9—C10	−0.1 (5)
Se1—P2—C12—C13	7.0 (4)	C8—C9—C10—C11	0.3 (5)
Se1—P2—C12—C17	−174.5 (3)	C9—C10—C11—C6	−0.4 (5)
O1—P2—C12—C13	−123.0 (3)	P2—C12—C13—C14	179.1 (3)
O1—P2—C12—C17	55.5 (4)	C17—C12—C13—C14	0.5 (6)
C6—P2—C12—C13	134.8 (3)	P2—C12—C17—C16	−178.3 (3)
C6—P2—C12—C17	−46.7 (4)	C13—C12—C17—C16	0.3 (6)
P2—O1—C1—O2	15.0 (5)	C12—C13—C14—C15	−1.3 (6)
P2—O1—C1—C2	−162.2 (2)	C13—C14—C15—C16	1.3 (6)
O1—C1—C2—C3	68.6 (4)	C14—C15—C16—C17	−0.5 (6)
O1—C1—C2—C4	−51.4 (4)	C15—C16—C17—C12	−0.3 (7)
O1—C1—C2—C5	−174.0 (3)		

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