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Section 29 (1)  
Regulation 3.1 (2)

AUSTRALIA

Patents Act 1990

## NOTICE OF ENTITLEMENT

We, Hoechst-Schering Agrevo GmbH of Abraham-Lincoln-Strasse 7, D-65189 Wiesbaden, Germany being the applicant in respect of Application No. 18113/95 state the following:-

The Person nominated for the grant of the patent has entitlement from the actual inventors under German Inventorship Law.

The person nominated for the grant of the patent is the applicant of the basic application listed on the patent request form.

The basic application listed on the request form is the first application made in a Convention country in respect of the invention.

HOECHST-SCHERING AGREVO GMBH  
By our Patent Attorneys,  
WATERMARK PATENT & TRADEMARK ATTORNEYS

2 June 1997

  
.....  
Karen J. Sinclair  
Registered Patent Attorney



AU9518113

(12) PATENT ABRIDGMENT (11) Document No. AU-B-18113/95  
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 681013

- (54) Title  
AQUEOUS ENDOSULPHANE SUSPENSION CONCENTRATES
- International Patent Classification(s)  
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- (87) PCT Publication Number : WO95/23508
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- (56) Prior Art Documents  
EP 224845  
DE 3210869

(57) Claim

1. An aqueous suspension concentrate of endosulfan, endosulfan, and comprising a surfactant combination of a neutralized phosphoric ester based on an ethoxylated alkylphenol and an ethoxylated alkylaryl- and alcohol phosphate ester.
10. A method of crop protection comprising applying an effective amount of an endosulfan concentrate as claimed in any one of claims 1 to 7 to crops or soils requiring protection.

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AOJP DATE 02/11/95 PCT NUMBER PCT/EP95/00641



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<p>(51) Internationale Patentklassifikation<sup>6</sup> : A01N 43/24 // (A01N 43/24, 25:30, 25:04)</p>	<p>A1</p>	<p>(11) Internationale Veröffentlichungsnummer: <b>WO 95/23508</b> (43) Internationales Veröffentlichungsdatum: 8. September 1995 (08.09.95)</p>
<p>(21) Internationales Aktenzeichen: PCT/EP95/00641 (22) Internationales Anmeldedatum: 22. Februar 1995 (22.02.95) (30) Prioritätsdaten: P 44 06 629.5 1. März 1994 (01.03.94) DE (71) Anmelder (für alle Bestimmungsstaaten ausser US): HOECHST SCHERING AGREVO GMBH [DE/DE]; Mirastrasse 54, D-13509 Berlin (DE). (72) Erfinder; und (75) Erfinder/Anmelder (nur für US): FRISCH, Gerhard [DE/DE]; Westerwaldstrasse 7, D-61273 Wehrheim (DE). MAIER, Thomas [DE/DE]; Kapellenstrasse 16, D-65710 Hofheim (DE).</p>	<p>(81) Bestimmungsstaaten: AU, CN, HU, JP, KR, KZ, MX, RU, UA, US, europäisches Patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  Veröffentlicht Mit internationalem Recherchenbericht.  <b>68 10 13</b></p>	
<p>(54) Title: AQUEOUS ENDOSULPHANE SUSPENSION CONCENTRATES (54) Bezeichnung: WÄSSRIGE SUSPENSIONSKONZENTRATE VON ENDOSULFAN (57) Abstract The present invention concerns liquid endosulphane formulations in the form of suspension concentrates containing a surfactant combination consisting of an ethoxylated alkylphenol-based phosphoric acid ester and an ethoxylated alkylaryl and alcohol phosphate ester. The invention further concerns a process for producing said concentrates and their use in pest control. (57) Zusammenfassung Die vorliegende Erfindung betrifft Flüssigformulierungen von Endosulfan in Form von Suspensionskonzentraten, die eine Tensidkombination aus einem Phosphorsäureester auf Basis eines ethoxylierten Alkylphenols und einem ethoxylierten Alkylaryl- und Alkoholphosphatester enthalten, Verfahren zu ihrer Herstellung und ihre Verwendung im Pflanzenschutz.</p>		

Hoechst Schering AgrEvo GmbH AGR 94/M 207 Dr.WS/PP

Description

Aqueous suspension concentrates of endosulfan

5 The present invention relates to liquid formulations of endosulfan in the form of suspension concentrates.

10 It is known that endosulfan [1,4,5,6,7,7-hexachloro-8,9,10-trinorborn-5-en-2,3-ylenebismethylene sulfite] is an active substance having an insecticidal activity (see US Patent 2 799 685). The active substance may be formulated in various ways, for example as an emulsifiable concentrate (EC), wettable powder (WP), water-dispersible granules (WG), concentrated aqueous emulsion (EW) and, finally, also as a suspension concentrate (SC). The advantages of SC formulations are, mainly, no development  
15 of dust and the absence of flammable solvents, and this latter fact can have positive effects mainly on the dermal toxicity to the user.

20 US Patent 4 804 399 discloses an SC formulation of endosulfan which, in addition to the active substance, comprises the alkali metal salt of a sulfosuccinic monoester and an alkali metal salt of a lignosulfonic acid in a mixture with a swellable alkaline earth metal silicate. This SC formulation is relatively viscous (> 400 mPas). Moreover, it comprises auxiliaries which do  
25 not meet the Environmental Protection Agency (EPA) requirements for auxiliaries (Exempt from tolerance under EPA Regulation 40 CFR 180.1001, c, d).

30 It must therefore be considered as surprising that it was possible to find a surfactant combination of auxiliaries which conform with EPA requirements, resulting in a stable SC formulation of endosulfan of particularly low viscosity. Moreover, these aqueous SC formulations cover a wide range of concentrations of suspended active

substance, which is even more surprising, bearing in mind the high specific gravity of endosulfan ( $1.8 \text{ g/cm}^3$ ), which is markedly higher than in the case of similar active substances for SC formulations, such as isoproturon ( $1.2 \text{ g/cm}^3$ ), linuron ( $1.5 \text{ g/cm}^3$ ) and carbendazim ( $1.5 \text{ g/cm}^3$ ).

The invention therefore relates to aqueous suspension concentrates of endosulfan comprising a surfactant combination of (1) a neutralized phosphoric ester based on an ethoxylated alkylphenol and (2) an ethoxylated alkylaryl- and alcohol phosphate ester, the concentration of active substance preferably being 50 to 650 g/l, in particular 300 to 500 g/l.

The combination according to the invention of the above-mentioned surfactant component (1), which is commercially available as <sup>®</sup>Emcol CS 1361 (Witco Corp.), with the abovementioned surfactant component (2) having preferably 9EO, which is commercially available as <sup>®</sup>Soprophor PA19 (Rhône Poulenc), is of utmost importance for the grindability in the preparation of the concentrate according to the invention and for its storage stability. This surfactant mixture, in which the ratio by weight of components (1) and (2) can vary from 100:1 to 1:100, preferably 10:1 to 1:10, in particular 3:1 to 1:3, moreover clearly suppresses crystal growth, which additionally contributes to the stability of this SC formulation over storage times of  $\approx 3$  months at temperatures of from  $-10^\circ\text{C}$  to  $+50^\circ\text{C}$ .

The surfactant combination content is preferably 1 to 30% by weight, in particular 2 to 15% by weight.

Further additives or auxiliaries, preferably selected from the group of the antifoams, antifreeze agents, alkaline earth metal silicates, thickeners, preservatives, wetting agents and dispersants, may be added to the SC formulation according to the invention.

The SC formulation preferably additionally comprises 0.2 to 3% by weight of antifoam, 0 to 12% by weight of antifreeze agent, 0.5 to 10% by weight of alkaline earth metal silicate, 0 to 0.2% by weight of thickener, 0 to 2%  
5 by weight of preservative and 0 to 3% by weight of customary wetting agents and dispersants.

Examples of customary wetting agents and dispersants are polyethoxylated alkylphenols, polyethoxylated fatty alcohols, tridecyl alcohol polyglycol ether (<sup>®</sup>Genapol  
10 X-080), alkyl- or alkylphenylsulfonates, sodium ligno-sulfonate, sodium 2,2'-dinaphthylmethane-6,6'-disulfonate, sodium dibutyl-naphthalenesulfonate or sodium oleylmethyltauride.

Examples of suitable swelling agents are swellable aluminosilicates or swellable polysaccharides, such as those  
15 which are prepared by fermentation of carbohydrates by means of Xanthomonas microorganisms, such as <sup>®</sup>Kelzan.

Other formulation auxiliaries which may be added are antifoams based on tributyl phosphate or based on silicone, such as dialkylpolysiloxanes, and antifreeze agents  
20 such as ethylene glycol, propylene glycol, glycerol, in particular propylene glycol, safeners such as, for example, urea, and customary preservatives such as, inter alia, benzoic acid, sorbic acid, formaldehyde, and traces  
25 of fungicidal active substances.

The invention also relates to a process for the preparation of the suspension concentrate according to the invention, which comprises stirring the active substance in an aqueous solution or suspension of the formulation  
30 auxiliaries and subsequently comminuting the resulting coarse suspension by grinding, if appropriate by grinding in a corundum mill or toothed-disk mill to finenesses of approximately 200 microns, and subsequently in ball mills or sand mills until the particle sizes in the suspension  
35 are 0.1 to 10 microns, preferably below 5 microns. The

particle sizes can be determined by means of a disk centrifuge or a Coulter counter apparatus.

5 The viscosity values of the SC formulations according to the invention are 40-60 mPas at speed 15 and 60-100 mPas at speed 1. In comparison, those of the SC formulations disclosed in US Patent 4 804 399 are 130-180 mPas at speed 15 and 610-660 mPas at speed 1 (all these values were determined at 20°C using a <sup>®</sup>Rheomat 115 rotary viscometer by Contraves). Due to the low viscosity, the  
10 spontaneity of these SC formulations when diluted with water is outstanding.

The invention also relates to a method of controlling harmful insects, which comprises applying an effective amount of the abovementioned SC concentrate in the form  
15 of an aqueous dilution to these harmful insects or to the plants and soils infested with them, and to the use of the SC concentrate in crop protection.

The compositions according to the invention are simply applied by diluting the suspension concentrates with the  
20 desired amount of water, stirring the mixture briefly and applying to the plant. The spray mixtures obtained from the suspension concentrates according to the invention are distinguished from the spray mixtures prepared with wetttable powders or emulsifiable concentrates in particular by the uniform distribution of the active substance,  
25 which is retained even after standing for 24 hours.

The present invention is illustrated by the examples which follow, which are compiled in Table I, without limiting the invention thereto. Table II lists examples  
30 in which a component of the surfactant mixture according to the invention was replaced and which are not storage-stable.

Table I (Percentages are by weight)

	1	2	3	4	5	6	7	8	9	10
Endosulfan	39.1	34.44	30.75	39.1	39.1	39.1	35.0	30.8	25.6	43.5
®Emcol CS 1361	4.0	3.36	3.00	2.0	4.0	4.0	3.6	3.5	3.0	5.0
®Soprophor PA-19	2.0	1.68	1.50	2.0	2.0	2.0	1.8	1.75	1.5	2.5
®Darvan No. 3	1.0	0.84	0.75	1.0	1.0	1.0	1.0	1.0	1.0	1.0
®Hostapon T	0.5	0.42	0.38	0.5	0.5	0.5	0.5	0.5	0.5	0.5
®Attapulgate Select 615	2.0	3.68	3.50	-	3.0	5.0	2.0	2.5	4.0	3.0
®Rhodorsil 5020	2.0	1.68	1.50	2.0	2.0	2.0	2.0	2.0	2.0	2.0
®Propylenglykol	8.0	6.72	6.00	8.0	8.0	7.0	8.0	8.0	10.0	9.0
®Rhodopol 23	-	-	-	0.1	-	-	-	-	-	-
Preservative	-	-	-	0.1	-	-	-	-	-	-
Water to 100%										

®Hostapon T (Hoechst AG) = sodium oleyl-N-methyltauride

®Attapulgate Select 615 (Oil Dri Corp. USA) = aluminum magnesium silicate equipped with hydrophobic properties

®Rhodorsil 5020 (Rhone Poulenc) = polydimethylsiloxane

®Darvan No. 3 (Vanderbilt Corp. USA) = sodium salt of a polymerized substituted alkylaryl-sulfonic acid with an inert inorganic suspending agent.

Table II (percentages are by weight)

	1	2	3	4	5	6	7
Endosulfan	39.1	39.1	39.1	39.1	39.1	39.1	39.1
®Emcol CS 1361	4.0	4.0	4.0	4.0	4.0	4.0	2.0
®Nekal BX	-	2.0	2.0	-	-	-	-
®Arkopal N 100	-	-	-	2.0	-	-	-
®Soprophor 860 P	-	-	-	-	2.0	-	-
®Witco ECD 1742	-	-	-	-	-	2.0	-
®Darvan No. 3	1.0	1.0	1.0	1.0	1.0	1.0	1.0
®Hostapon T	0.5	0.5	0.5	0.5	0.5	0.5	0.5
®Rhodorsil 5020	2.0	2.0	2.0	2.0	2.0	2.0	2.0
®Attapulgate Select 615	2.0	2.0	-	2.0	2.0	2.0	-
®Bentone EW	-	-	0.5	-	-	-	-
®Rhodopol 23	-	-	-	-	-	-	0.2
Propylene glycol	8	8	8	8	8	8	8
Water to 100%							

For the trademarks which are not described here in detail, see: McCutcheon's Emulsifiers & Detergents, Vol. 1, International Edition

Patent claims:

1. An aqueous suspension concentrate of endosulfan, endosulfan, and comprising a surfactant combination of a neutralized phosphoric ester based on an ethoxylated alkylphenol and an ethoxylated alkylaryl- and alcohol phosphate ester.  
5
2. A concentrate as claimed in claim 1 comprising 50 to 650 g of endosulfan/l.
3. A concentrate as claimed in claim 1 or 2, in which the ratio by weight of the two components of the surfactant combination defined in claim 1 is 100:1 to 1:100.  
10
4. A concentrate as claimed in any of claims 1 to 3 comprising 1 to 30% by weight of the surfactant combination.  
15
5. A concentrate as claimed in any of claims 1 to 4 comprising 2 to 15% by weight of the surfactant combination.
6. A concentrate as claimed in any of claims 1 to 5 comprising further additives or auxiliaries, preferably selected from the group of the antifoams, antifreeze agents, alkaline earth metal silicates, thickeners, preservatives, wetting agents and dispersants.  
20
7. A concentrate as claimed in any of claims 1 to 6, additionally comprising 0.2 to 3% by weight of antifoam, 0 to 12% by weight of antifreeze agent, 0.5 to 10% by weight of alkaline earth metal silicate, 0 to 0.2% by weight of thickener, 0 to 2% by weight of preservative and 0 to 3% by weight of customary wetting agent and dispersant.  
25  
30



8. A process for the preparation of an endosulfan concentrate as claimed in any of claims 1 to 6, which comprises stirring the endosulfan and a surfactant combination of a neutralized phosphoric ester based on an ethoxylated alkylphenol and an ethoxylated alkylaryl and alcohol phosphate ester in an aqueous solution or suspension of formulation auxiliaries and subsequently comminuting the resulting coarse suspension by grinding.

9. A method of controlling harmful insects, which comprises applying an effective amount of an endosulfan concentrate as claimed in any one of claims 1 to 7 in the form of an aqueous dilution to these harmful insects or to the plants and soils infested with them.

10. A method of crop protection comprising applying an effective amount of an endosulfan concentrate as claimed in any one of claims 1 to 7 to crops or soils requiring protection.

DATED THIS 2ND DAY OF JUNE, 1997

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KJS:TJ  
DOC 14 AU1417295.WPC



**INTERNATIONAL SEARCH REPORT**

International Application No  
PCT/EP 95/00641

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 6 A01N43/24 //(A01N43/24,25:30,25:04)

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	DE,A,32 10 869 (KAO CORP) 14 October 1982 see claims 1,6,11 see page 4, line 8 - line 16 see page 5, line 31 - page 6, line 25 see page 7, line 31 - page 8, line 2 see page 8, line 19 ---	1-10
Y	EP,A,0 224 845 (HOECHST AG) 10 June 1987 see page 1, line 3 - line 6 see page 2, line 28 - line 31 see page 3, line 12 - line 18 see page 6, line 30 --- -/--	1-10

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

\* Special categories of cited documents :

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- \*P\* document published prior to the international filing date but later than the priority date claimed

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- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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- \* & \* document member of the same patent family

Date of the actual completion of the international search

16 May 1995

Date of mailing of the international search report

09.06.95

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## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP 95/00641

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE,A,37 02 604 (SIDECO CHEMIE DR SCHIRM GMBH) 11 August 1988 see claim 5 see page 2, line 44 - line 48 see page 2, line 67 see page 3, line 41 - line 43 see page 4, line 6 - line 9 ----	1-10
A	DE,A,33 02 648 (HOECHST AG) 2 August 1984 see claims 1,4,6 see page 7, line 21 - page 8, line 9 ----	1-10
A	EP,A,0 110 174 (HOECHST AG) 13 June 1984 see the whole document & US,A,4 804 399 cited in the application -----	1-10

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 95/00641

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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		US-A- 4804399	14-02-89
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INTERNATIONALER RECHERCHENBERICHT

Internationales Aktenzeichen

PCT/EP 95/00641

A. KLASSIFIZIERUNG DES ANMELDUNGSGEGENSTANDES  
 IPK 6 A01N43/24 //(A01N43/24, 25:30, 25:04)

Nach der Internationalen Patentklassifikation (IPK) oder nach der nationalen Klassifikation und der IPK

B. RECHERCHIERTE GEBIETE

Recherchiertes Mindestprüfstoff (Klassifikationssystem und Klassifikationssymbole)

IPK 6 A01N

Recherchierte aber nicht zum Mindestprüfstoff gehörende Veröffentlichungen, soweit diese unter die recherchierten Gebiete fallen

Während der internationalen Recherche konsultierte elektronische Datenbank (Name der Datenbank und evtl. verwendete Suchbegriffe)

C. ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie*	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
Y	DE,A,32 10 869 (KAO CORP) 14.Oktober 1982 siehe Ansprüche 1,6,11 siehe Seite 4, Zeile 8 - Zeile 16 siehe Seite 5, Zeile 31 - Seite 6, Zeile 25 siehe Seite 7, Zeile 31 - Seite 8, Zeile 2 siehe Seite 8, Zeile 19 ---	1-10
Y	EP,A,0 224 845 (HOECHST AG) 10.Juni 1987 siehe Seite 1, Zeile 3 - Zeile 6 siehe Seite 2, Zeile 28 - Zeile 31 siehe Seite 3, Zeile 12 - Zeile 18 siehe Seite 6, Zeile 30 ---	1-10

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Weitere Veröffentlichungen sind der Fortsetzung von Feld C zu entnehmen

Siehe Anhang Patentfamilie

\* Besondere Kategorien von angegebenen Veröffentlichungen :

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\*Z\* Veröffentlichung, die Mitglied derselben Patentfamilie ist

Datum des Abschlusses der internationalen Recherche

16.Mai 1995

Absenddatum des internationalen Recherchenberichts

09.06.95

Name und Postanschrift der Internationalen Recherchenbehörde  
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Bevollmächtigter Bediensteter

Lamers, W

## INTERNATIONALER RECHERCHENBERICHT

Ink. Internationales Aktenzeichen

PCT/EP 95/00641

C.(Fortsetzung) ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
A	DE,A,37 02 604 (SIDECO CHEMIE DR SCHIRM GMBH) 11.August 1988 siehe Anspruch 5 siehe Seite 2, Zeile 44 - Zeile 48 siehe Seite 2, Zeile 67 siehe Seite 3, Zeile 41 - Zeile 43 siehe Seite 4, Zeile 6 - Zeile 9 ----	1-10
A	DE,A,33 02 648 (HOECHST AG) 2.August 1984 siehe Ansprüche 1,4,6 siehe Seite 7, Zeile 21 - Seite 8, Zeile 9 ----	1-10
A	EP,A,0 110 174 (HOECHST AG) 13.Juni 1984 siehe das ganze Dokument & US,A,4 804 399 in der Anmeldung erwähnt -----	1-10

# INTERNATIONALER RECHERCHENBERICHT

Angaben zu Veröffentlichungen, die zur selben Patentfamilie gehören

Inte: Internationales Aktenzeichen

PCT/EP 95/00641

Im Recherchenbericht angeführtes Patentdokument	Datum der Veröffentlichung	Mitglied(er) der Patentfamilie	Datum der Veröffentlichung
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		CA-A- 1282608	09-04-91
		JP-A- 62132802	16-06-87
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DE-A-3702604	11-08-88	KEINE	
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DE-A-3302648	02-08-84	AU-B- 562412	11-06-87
		AU-A- 2383784	02-08-84
		CA-A- 1227352	29-09-87
		EP-A, B 0117999	12-09-84
		US-A- 4594096	10-06-86
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EP-A-0110174	13-06-84	DE-A- 3240862	10-05-84
		AU-B- 562459	11-06-87
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		CA-A- 1226210	01-09-87
		JP-C- 1681743	31-07-92
		JP-B- 3048881	25-07-91
		JP-A- 59098002	06-06-84
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