



Industria y Comercio

SUPERINTENDENCIA



Delegatura de propiedad Industrial

División de Nuevas Creaciones

Organización CSA Ltda

PCT
SOLICITUD

PATENTE DE INVENCION

2/5

21. EXPEDIENTE No.

06-54552

54. TÍTULO Composiciones de Ionómeros Estéreo-Reliables
curables por energía Solubles en agua

51. CLASIFICACIÓN INTERNACIONAL C08F 8/00 8/44

71. SOLICITANTE Sun Chemical Corporation

DOMICILIO Fort Lee, NJ USA.

74. APODERADO Dilia Maria Rodriguez d'Almeida

22. BOGOTÁ, D. C., junio 6/06.
707099

FORMULARIO ÚNICO DE SOLICITUD DE PATENTE
PCT
ENTRADA EN FASE NACIONAL

1) SOLICITUD DE:

☒ Patente de invención ☐ Patente de Modelo de Utilidad

☒ Capitulo I. ☐ Capitulo II.

2) SOLICITANTE
(71)

Nombre: Sun Chemical Corporation

Dirección: 222 Bridge Plaza South,
Fort Lee, NJ 07024, Estados Unidos

Nacionalidad o domicilio: New
Jersey, Estados Unidos

Lugar de constitución: New Jersey
Estados Unidos

IDENTIFICACIÓN

C.C. ☐ NIT: ☒

C.E. ☐ OTRO ☐

NÚMERO Cual _____

Teléfono: Fax:
E. Mail:

3) REPRESENTANTE
O APODERADO

Nombre: DILIA MARÍA RODRÍGUEZ
D'ALEMAN

Dirección: Carrera 11 N°. 86-53 Piso 6
Bogotá D.C.

Teléfono: 6181088 Fax: 6350824
E. Mail: info@clarkemodet.com.co

IDENTIFICACIÓN

C.C. ☒ NIT: ☐

C.E. ☐ OTRO ☐

NÚMERO: 41.654.882
TP: 20824 CSJ

4) INVENTOR (ES)
(72)

Nombre: 1. LINZER, Volker; 2. JONES, Richard, M.
Dirección: 1. 417 Seventh Street, Carlstadt, NJ 07072 USA; 2.
161 Greendale Drive, Oak Ridge, NJ 07438 USA

Nacionalidad o Domicilio: Todos los anteriores,
Estadounidenses

5) PCT (86)

Solicitud Internacional No. PCT/US2004/037222 Fecha: Nov. 5, 2004

Publicación Internacional No. WO 2005/047345 A1 Fecha: Mayo 26, 2005

6) Título (54) COMPOSICIONES DE IONÓMEROS ESTÉREO-RETICULABLES
CURABLES POR ENERGÍA SOLUBLES EN AGUA

7) Clasificación Internacional: CO8F 8/00, 8/44

8) PRIORIDAD

SI ☒ NO ☐

33) País de Origen

Estados Unidos

(32) Fecha

Nov. 6, 2003

(31) Número de
Solicitud

10/702,313

9) Comprobante de pago N°. 06-40,121 de Mayo 26 de 2006

Instrucciones para el diligenciamiento del presente formulario. Ver reverso de esta pagina

10)

ANEXOS

- ☒ Comprobante de pago de la tasa de presentación de la solicitud
- ☐ Comprobante de pago por reivindicación de prioridad
- ☒ Documento que acredita la existencia y representación legal cuando el solicitante sea persona Jurídica
- ☒ Poderes, si fuera el caso
- ☐ Documento de cesión del inventor al solicitante o a su causante
- ☐ Declaraciones
- ☒ Copia de la solicitud Internacional. (Resumen, descripción, reivindicación, Dibujos).
- ☒ Traducción de la solicitud internacional al castellano. (Resumen, descripción, reivindicación, Dibujos)
- ☐ Copia del examen preliminar efectuado por la IPEA
- ☐ De ser el caso, copia del contrato de acceso
- ☐ De ser el caso, documento que acredite la licencia o autorización de uso de conocimientos tradicionales de las comunidades indígenas
- ☐ De ser el caso, certificado de depósito del material biológico
- ☐ Arte final 12 x 12 y 6 x 6 cm
- ☐ De ser el caso, información sobre otras solicitudes de patente o títulos obtenidos en el extranjero por el mismo titular o su causante, relacionados parcial o totalmente con la invención de esta solicitud.
- ☐ Otros:
 1. Reporte de búsqueda
 2. Opinión escrita
 3. Solicitud PCT
 4. PCT documento de prioridad
 5. Países designados

11) FIGURA CARACTERÍSTICA:

12) Solicitud concesión de la patente,

NOMBRE: DILIA RODRÍGUEZ D'ALEMAN

FIRMA:

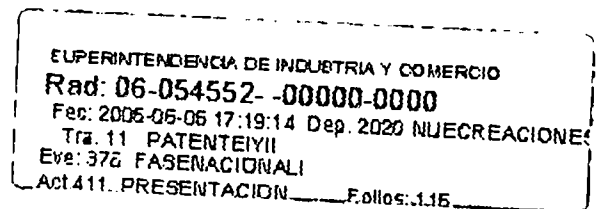
C.C. 41.654.882 de Bogota TP 20824 CSJ

Instrucciones para el diligenciamiento del presente formulario. Ver reverso de esta pagina

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

NIT : 800176089-2

RECIBO OFICIAL DE CAJA : 06 - 40,121
FECHA : MAYO 26 DE 2006



***** CONSIGNACION *****

DEPOSITANTE	TIPO PAGO	BANCO	CUENTA	No. PAGO	FECHA PGO	Vr. PAGO
CLARKE MODET Y CO. COLOMB	CONSIGNACION	BANCO POPULAR	050-00110-6	49045937	26/05/2006	12,022,000.00

***** CONCEPTO *****

CANT.	RENTISTICO	CONCEPTO	TOTAL CONCEPTO
1	50005-01-01	SOLICITUDES	463,000.00
1		TRAMITES DE SOL. DE PATENTE DE IN	463,000.00
		TOTAL :	\$ 463,000.00

SON: CUATROCIENTOS SESENTA Y TRES MIL PESOS

RESPONSABLE : _____

RECIBO DE CAJA APLICADO AL EXPEDIENTE No. _____

TRADUCCIÓN OFICIAL No. 01/074 de un documento escrito en inglés, el cual para su identificación se sella con el sello del traductor al tiempo con la presente. Esta traducción ha sido preparada por Juan Pablo Piedrahita Aguilera, Traductor e Intérprete Oficial debidamente reconocido de conformidad con el Certificado de Idoneidad Profesional No. 0045 expedido por la Universidad Nacional de Colombia.

(Se traduce un Certificado de Notarial, Legalización y Apostilla escritos en inglés, adjuntos a un poder otorgado por la compañía SUN CHEMICAL CORPORATION, escrito en inglés y español, con fecha Febrero 13, 2001.)

CERTIFICADO NOTARIAL (Cuando el Solicitante es una Sociedad)

(Poderes otorgados en El Salvador, México, Estados Unidos de América y Venezuela)

Expedido hoy 13 de Febrero, 2001 el suscrito Notario CERTIFICA que:

- 1) Melvin M. Cox (nombre de la persona que firma el poder), mayor de edad y domiciliada en Fort Lee, New Jersey, suscribió el anterior documento.
- 2) El otorgante le es conocido personalmente y tiene facultad legal para firmar el documento.
- 3) El otorgante ha suscrito el anterior documento en su calidad de Vicepresidente de la persona jurídica Sun Chemical Corporation.

JUAN PABLO PIEDRAHITA AGUILERA
TRADUCTOR E INTÉRPRETE OFICIAL
C. IDONEIDAD 045
UNIVERSIDAD NACIONAL DE COLOMBIA



4) La mencionada persona jurídica con domicilio comercial en 222 Bridge Plaza South, Fort Lee, New Jersey, está debidamente constituida, tiene actualmente existencia legal y el fin para el cual se otorga este poder se encuentra dentro de su objeto social.

5) El otorgante tiene en efecto la idoneidad referida y su representación es legal y está autorizado para otorgar este poder.

6) Fundamento las certificaciones 3), 4) y 5) en los documentos auténticos que para tal fin me fueron exhibidos.

(Firmado) Elizabeth C. Reyes

Notario Público

(Sello Notarial a Tinta: S. Elizabeth C. Reyes, Notario Público para New Jersey, Mi Comisión Expira Febrero 13, 2002)

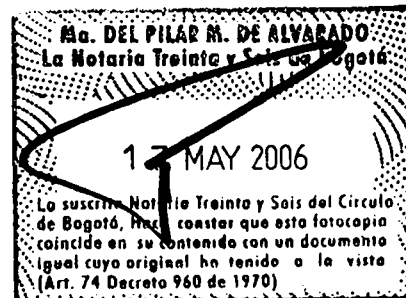
(Sello Notarial Redondo en Relieve)

Nota: El Consulado de Colombia debe autenticar la firma del notario público.

ESTADO DE NEW JERSEY,

CONDADO DE BERGEN} ss. :

Yo, Kathleen A. Donovan, Escribano del Condado de Bergen, (el mismo siendo una Corte de Registro de dicho Condado, que tiene por ley un sello), POR EL PRESENTE CERTIFICO QUE Elizabeth C. Reyes, cuyo nombre está suscrito en el certificado de reconocimiento, prueba o declaración juramentada adjunto, era



6

en el momento de recibir tal reconocimiento, prueba o declaración juramentada un NOTARIO PUBLICO debidamente comisionado, juramentado y residente en dicho Estado y como tal NOTARIO PÚBLICO, es un funcionario de dicho Estado debidamente autorizado por sus leyes para tomarlos y certificarlos, asó como para tomar y certificar las pruebas y reconocimientos de escrituras para traspasos de tierra, tenencias o herencias, y otros instrumentos por escrito, para registro en dicho Estado; y que dicho reconocimiento está debidamente firmado y tomado conforme a las leyes de dicho Estado y que estoy bien familiarizado con su caligrafía y verdaderamente creo que la firma en el certificado adjunto es su firma auténtica.

Además certifico que no se requiere por las leyes de este Estado que la impresión del sello de dicho NOTARIO PÚBLICO esté en los archivos de mi oficina.

EN TESTIMONIO DE LO CUAL firmo y estampo mi sello oficial hoy 13 de Febrero de 2001 A.D.

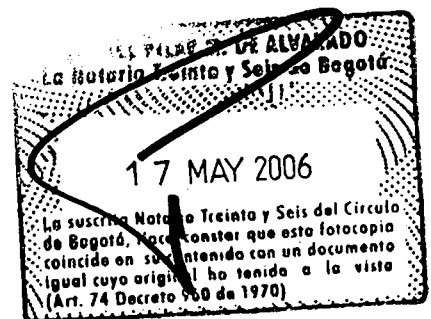
(Firmado) Kathleen A. Donovan, Escribano

(Sello en Relieve del Condado de Bergen, NJ)

APOSTILLA

(Convención de la Haya de Octubre 5 de 1961)

1. País: Estados Unidos de América
Este documento público
2. Ha sido firmado por Kathleen A. Donovan



3. Actuando en calidad de Escribano del Condado
4. Lleva el sello/estampilla de Condado de Bergen

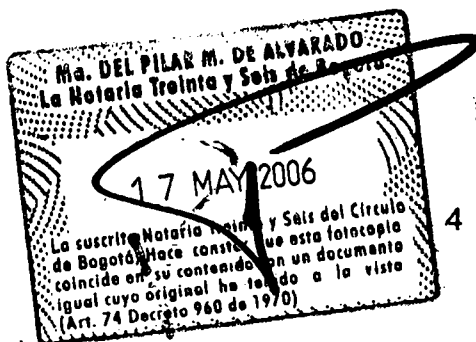
CERTIFICADO

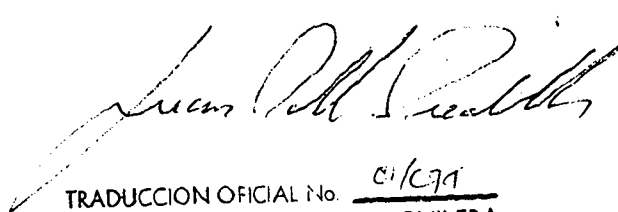
5. En Trenton, New Jersey 6. Hoy 21 de Febrero, 2001
7. Por Peter R Lawrence, Tesorero de New Jersey
8. Certificado Número A128014
9. (SELLO)
- (Sello Redondo de Oblea en Relieve)
10. Firma: (Firmado) Peter R Lawrence

Certificado de Apostilla No. A128014 por el Tesorero de New Jersey, de fecha
Febrero 21, 2001.

ES TRADUCCIÓN FIEL Y COMPLETA de la cual se deja copia en los archivos de
esta oficina para su confrontación y a la cual me remito.

Bogotá, Marzo 8 de 2001.



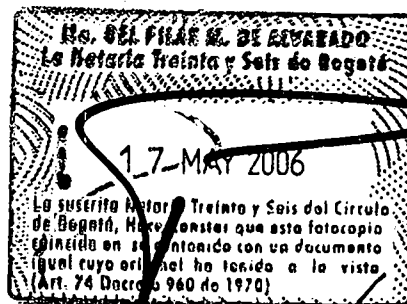

TRADUCCION OFICIAL No. 01/070
JUAN PABLO PIEDRAHITA AGUILERA
TRADUCTOR E INTERPRETE OFICIAL
C. IDONEIDAD 045
UNIVERSIDAD NACIONAL DE COLOMBIA

MINISTERIO DE
RELACIONES EXTERIORES

NO SE ASUME
RESPONSABILIDAD DEL TEXTO

245262
Juan Presabity
CUYA FIRMA APARECE EN EL
DOCUMENTO DESEMPENA
LAS FUNCIONES INDICADAS

2004 JUL 28 A 11:54
ues
JEFE DE LEGALIZACIONES
BOGOTÁ D.C.



7.

P400

APOSTILLE

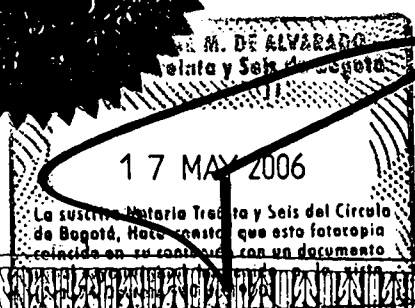
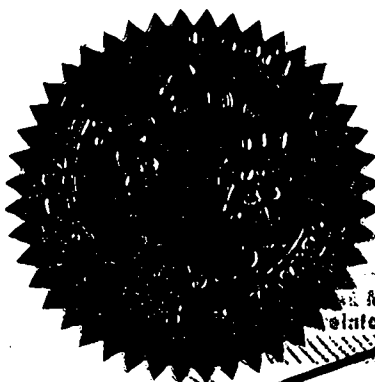
(CONVENTION DE LA HAYE DU 5 OCTOBRE 1961)

1. COUNTRY: UNITED STATES OF AMERICA
2. THIS PUBLIC DOCUMENT HAS BEEN SIGNED BY:
KATHLEEN A DONOVAN
3. ACTING IN THE CAPACITY OF:
COUNTY CLERK
4. BEARS THE SEAL/STAMP OF:
COUNTY OF BERGEN

CERTIFIED

5. AT TRENTON, NEW JERSEY
6. THE 21ST DAY OF FEBRUARY 2001
7. BY: Peter R Lawrance, NEW JERSEY TREASURER
8. NO. A128014
9. SEAL/STAMP:
10. SIGNATURE

Peter R Lawrance



TRADUCCION OFICIAL No. 61/671
JUAN PABLO PIEDRAHITA AGUILERA
TRADUCTOR E INTERPRETE OFICIAL
C. IDONEIDAD 045
UNIVERSIDAD NACIONAL DE COLOMBIA

Clarke, Modet & Co. Colombia Ltda.

PROPIEDAD INDUSTRIAL y DERECHOS DE AUTOR

Apartado Aéreo 92397
Santafé de Bogotá, Colombia

PODER

Yo (nosotros), abajo firmante(s)

Melvin M. Cox

en mi (nuestro) carácter de representante(s) legales de

Sun Chemical Corporation

una sociedad organizada y existente de acuerdo con las leyes de
Delaware

y en actual cumplimiento de su objeto social, con domicilio en

222 Bridge Plaza South
Fort Lee, NJ 07024 USA

por el presente instrumento confiero (conferimos) poder especial a favor de DILIA MARIA RODRIGUEZ D'ALEMAN, con Cédula de Ciudadanía No. 41.654.882 de Bogotá, y tp. 20824 y CLAUDIA LUCIA CARO RAMIREZ, con Cédula de Ciudadanía No. 40.017.374 de Tunja, y tp. 36448, con domicilio en Santa Fé de Bogotá, Colombia, para que actuando en su orden en nombre del (de la) otorgante le (la) representen ante cualquier entidad, autoridad y/o persona, especialmente ante las del orden Administrativo, Contencioso Administrativo, Judicial, Tribunales de Arbitramento y/o cualquier otro en lo referente a los siguientes asuntos:

- 1) Solicitar, tramitar, obtener y/o registrar cualquiera de las modalidades de Propiedad Industrial, tales como: patentes de invención, modelos de utilidad; diseños industriales, variedades vegetales; marcas, lemas, nombres y enseñas comerciales y denominaciones de origen; así como sus renovaciones, prórogas, transferencias, cambios de nombre y/o domicilio, contratos y licencias.
- 2) Solicitar, tramitar, obtener y/o registrar derechos de autor, en cualquiera de sus presentaciones así como la protección de secretos industriales y know-how.
- 3) Solicitar, tramitar, obtener y/o registrar registros sanitarios, permisos y/o licencias de funcionamiento; así como sus renovaciones o prórogas, transferencias, cambios de nombre y/o domicilio, contratos y licencias.
- 4) Ejercer acciones de oposición, observación, reclamos, reconsideración, reposición, apelación, cancelación administrativa o judicial, nulidad, protección al consumidor, concesión de licencias, protección de secretos industriales y demás acciones y recursos relacionados con derechos derivados de los asuntos arriba indicados y en contra de la competencia desleal, acciones derivadas de la obtención o explotación de licencias, acciones de indemnización de perjuicios. También podrá(n) intervenir en juicios civiles, comerciales, penales, contencioso-administrativos, tutelas y de policía; en acciones o juicios promovidos por el (la) mandante o en contra el (ella).
- 5) Registrar nombres de dominio ante la entidad competente.
- 6) En el ejercicio de este mandato los mandatarios arriba mencionados quedan facultados para: conciliar, cancelar, transigir, comprometer en árbitros, desistir, recibir y tomar posesión de la cosa objeto de litigio y renunciar o desistir a derechos concedidos o en curso de concesión, admitir los hechos del proceso.
- 7) Ratificar o no la agencia oficiosa.
- 8) Así mismo faculto (facultamos) a los mencionados apoderados para recibir notificaciones y designar apoderados judiciales o extrajudiciales, sustituir el presente poder y revocar las sustituciones que hicieren.

Dado y firmado en Fort Lee, NJ, USA

Mo. DEL PILAR M. DE ALMEIDA
La Notaria Tercera y Seis de Bogotá de

Feb 6 de 2001

17 MAY 2006

La suscrita Notaria Tercera y Seis del Circuito de Bogotá, Hace constar que esta fotocopia coincide en su contenido con un documento igual cuyo original ha venido a la vista (Art. 74 Decreto 960 de 1970)

POWER OF ATTORNEY

I (we), the undersigned

Melvin M. Cox

legal representative(s) of

Sun Chemical Corporation
an organization existing under the laws of

Delaware

and in present execution of its business activity, domiciled at

222 Bridge Plaza South
Fort Lee, NJ 07024 USA

do hereby grant POWER OF ATTORNEY to DILIA MARIA RODRIGUEZ D'ALEMAN, identification card No. 41.654.882 of Bogotá, and tp. 20824 and CLAUDIA LUCIA CARO RAMIREZ, tp. 36448, identification card No. 40.017.374 of Tunja, residing in Santa Fé de Bogotá, Colombia, to act in the order of their appointment in behalf of grantor before any entity, authority and/or natural or juridical person, specially before those Administrative, Administrative-Contentious, Judicial, arbitration courts, and/or any other, in all matters concerning the following:

- 1) To apply, process, obtain and/or register any of the modalities of Industrial Property, such as: Patents of Invention, Utility Models, Designs, vegetal varieties; Marks, Slogans, Trade Names and emblems and appellations of Origin; as well as their renewals, extensions, assignments, changes of name and/or domicile, agreements and licenses.
- 2) To apply, process, obtain and/or register copyrights of any kind as well as protection of industrial secrets or know-how.
- 3) To apply, process, obtain and/or register sanitary registrations, operating authorizations and/or licenses; as well as their renewals or extensions, assignments, changes of name and/or domicile, agreements and licenses.
- 4) To bring actions of opposition, observations, claims, reconsideration, appeal or legal remedies, cancellations, nullity, protection to the consumer, granting of licenses, protection of trade secrets and other actions related with rights derived from the above-mentioned matters and against the unfair competition, actions resulting from the obtention or exploitation of licenses, recovering damages actions. They can also intervene or participate in Civil, Commercial, Criminal or Contentious-Administrative Trials; the actions for writ mandamus, police actions; in actions, lawsuits or litigations instituted by the grantor or against him.
- 5) To register domain names before the competent entity.
- 6) To the effect of this mandate the above-named proxy are empowered to: conciliate, cancel, settle, submit to arbitration, withdraw and to receive and take possession of the litigation related object and waive or renounce the rights granted or in process of granting, admit the facts of the case.
- 7) To ratify or not the officious agency.
- 8) I (we) hereby appoint the afore mentioned attorneys with powers to receive notifications and to designate judicial and extrajudicial attorneys, substitute this Power of Attorney and to revoke such substitutions.

Granted and signed at Fort Lee, NJ, USA

on this 13th day of February of 2001

Melvin M. Cox

Firma / Signature

By: Melvin M. Cox, Vice President, Sun Chemical

CERTIFICADO NOTARIAL (Cuando el solicitante es SOCIEDAD)

(Poderes otorgados en: El Salvador, Estados Unidos, México y Venezuela)

A los _____ días del mes de _____
de _____, el Notario que suscribe DA FE de que:

1. _____
(nombre de la persona que firma el poder)

mayor de edad y domiciliado _____
_____ firmó de su puño
y letra el documento que antecede.

2. Conoce al (a la) otorgante y que este (a) está legalmente capacitado para el otorgamiento.

3. El otorgante ha firmado el referido documento en su carácter de _____
de la persona jurídica _____

4. La citada persona jurídica con sede en _____
_____ está legalmente constituida, que tiene actualmente existencia legal y que el acto para el cual se ha otorgado este poder está comprendido entre los que constituyen su objeto social.

5. El otorgante tiene efectivamente la representación que ostenta, que esta es legítima y que está facultado para otorgar este poder.

6. Las anteriores certificaciones de los puntos 3, 4 y 5, me constan porque he tenido a la vista los documentos auténticos.

Notario Público

NOTA: A continuación de lo anterior, el Consul de Colombia debe autenticar la firma del Notario Público.

CERTIFICADO NOTARIAL (Cuando el solicitante es INDIVIDUO)

A los _____ días del mes de _____
de _____, el Notario que suscribe DA FE de que:

1. _____
(nombre de la persona que firma el poder)

mayor de edad y domiciliado en _____
_____ firmó en su presencia el documento que antecede, conoce al otorgante y que este está legalmente capacitado para el otorgamiento.

Notario Público

NOTA: A continuación de lo anterior, el Consul de Colombia debe autenticar la firma del Notario Público.

Para todos los demás países el Señor Consul de Colombia o ante quien se otorgue debe expedir una certificación dejando constancia 1) de la existencia legal de la sociedad, 2) de que esta ejerce actualmente su objeto social, y 3) de que la persona o personas que otorgan el poder están facultadas para hacerla. El Notario Reintenta y Se documentos evidenciando esas circunstancias que acrediten tales circunstancias.

NOTARIAL CERTIFICATE (When the applicant is a CORPORATION)

(Powers of attorney granted in: El Salvador, Mexico, U.S.A. and Venezuela)

This 13th day of February 2001,
the undersigned Notary CERTIFIES that:

1. Melvin M. Cox
(name of the person who signs the power of attorney)

_____ of legal age and domiciled in Fort Lee, NJ
set his hand on the foregoing document.

2. The grantor is to him personally known and that he/she has legal capacity to execute the document.

3. The grantor has executed the foregoing document in his capacity of Vice President
of the juridical person Sun Chemical Corporation

4. The mentioned juridical person with place of business in 222 Bridge Plaza South, Fort L NJ
is duly organized, has actual legal existence and that the purposes for which the power of attorney is granted are within the scope of its corporate purposes.

5. The grantor has in fact the referred quality and that his/her representation is legal and that he/she is empowered to grant this power of attorney.

6. I have based the above certifications 3, 4 and 5, on the authentic documents which for such purpose were exhibited to me.

ELIZABETH C. REYES
A Notary Public of New Jersey
My Commission Expires February 13, 2002

Notary Public

NOTA: Then the Colombian Consul must legalize the signature of the Notary Public.

NOTARIAL CERTIFICATE (When the applicant is an INDIVIDUALS)

On this _____ day of _____
the undersigned Notary CERTIFIES that:

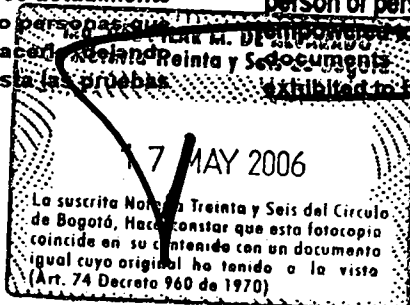
1. _____
(name of the person who signs the Power of Attorney)

_____ of legal age and domiciled at _____
_____ signed in his presence the foregoing document, the grantor is to him personally known and that he/she has legal capacity to execute the document.

Notary Public

NOTA: Then the Colombian Consul must legalize the signature of the Notary Public.

For all other countries the Colombian Consul or before who it is granted must issue a certification stating 1) the legal existence of the corporation, 2) that the corporate purposes are actually being accomplished, 3) that the person or persons who grant this Power of Attorney are duly qualified to do it. The Consul will certify also that the documents evidencing those circumstances were exhibited to him.



STATE OF NEW JERSEY,
COUNTY OF BERGEN) SS.:

I, Kathleen A. Donovan, Clerk of the County of Bergen
(the same being an office of Record of the aforesaid County,
having by law a seal)

DO HEREBY CERTIFY, That, Elizabeth C. Reyes
whose name is subscribed to the attached certificate of acknowledgement, proof or affidavit, was at the time of taking said
acknowledgement, proof or affidavit, a NOTARY PUBLIC, duly commissioned and sworn and residing in said State, and was,
as such NOTARY PUBLIC, an officer of said State duly authorized by the laws thereof to take and certify the same, as well as
to take and certify the proof and acknowledgement of deeds for the conveyance of land, tenements or hereditaments, and other
instruments in writing to be recorded in said State, and that the said acknowledgement is duly executed and taken according to
the laws of said State, and that I am well acquainted with his/her handwriting and verily believe the signature to the attached
certificate is his/her genuine signature.

And I do further certify that the impression of the seal of such NOTARY PUBLIC is not required by the laws of this
State to be filed in my office.

IN WITNESS WHEREOF, I have hereunto set my hand and affixed my official seal this

13

day of February

A.D., 20 01

Kathleen A. Donovan

Clerk

Deputy

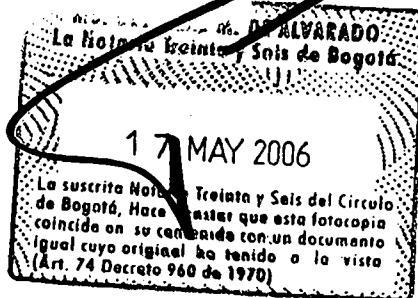
TRADUCCION OFICIAL No. 01/034

JUAN PABLO PIEDRAHITA AGUILERA

TRADUCTOR E INTERPRETE OFICIAL

C. IDONEIDAD 045

UNIVERSIDAD NACIONAL DE COLOMBIA





(43) International Publication Date
26 May 2005 (26.05.2005)

PCT

(10) International Publication Number
WO 2005/047345 A1

(51) International Patent Classification⁷: C08F 8/00, 8/44
(21) International Application Number:
PCT/US2004/037222

(22) International Filing Date:
5 November 2004 (05.11.2004)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
10/702,313 6 November 2003 (06.11.2003) US

(71) Applicant (for all designated States except US): SUN
CHEMICAL CORPORATION [US/US]; 222 Bridge
Plaza South, Fort Lee, NJ 07024 (US).

(72) Inventors; and
(75) Inventors/Applicants (for US only): LINZER, Linzer
[US/US]; 417 Seventh Street, Carlstadt, NJ 07072 (US).
JONES, Richard, M. [US/US]; 161 Greendale Drive, Oak
Ridge, NJ 07438 (US).

(74) Agent: PERSLEY, Sidney; 222 Bridge Plaza South, Fort
Lee, NJ 07024 (US).

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(54) Title: WATER SOLUBLE ENERGY CURABLE STEREO-CROSSLINKABLE IONOMER COMPOSITIONS

(57) Abstract: A homogenous, aqueous, energy curable, stereo cross linkable ionomer containing coating composition and a method
of preparing same.



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**WATER SOLUBLE ENERGY CURABLE STEREO-CROSSLINKABLE
IONOMER COMPOSITIONS**

FIELD OF THE INVENTION

This invention relates to the synthesis and use of water soluble energy curable stereo crosslinkable ionomers used in the manufacture of coated and printed materials.

BACKGROUND OF THE INVENTION

Ionomers are polymeric compounds carrying electronic charges within the polymer chain. Ionomers build outstanding properties like hardness and solvent resistance when two or more such polymeric chains create a ladder-type structure by salt formation. The ionic complexation of identical (e.g., bridged by a higher-valent counterion) or opposing charges (e.g., acid/base neutralization of amine-functional with carboxyl-functional polymeric species) on the polymer chains results in additional crosslinks, the number per unit volume of which determines the mechanical strength of the resulting solid. The more commonly used ionic complexation route typically proceeds through use of a more stable complex of the bridging counterion with a volatile material (e.g. ammonia) to allow blending in the liquid state followed by later crosslinking upon drying to a solid. The acid/base neutralization of two oppositely charged ionomers to create crosslinking, in contrast, is largely impractical due to the impossibility of blending these materials to generate anything other than an intractable crosslinked solid.

Description of Related Art

The photopolymerization approach to ionomer crosslinking is not novel. U.S. Patents 6,281,271 and 6,017,982 disclose the energy curing of ethylenically unsaturated ions to build covalent molecular weight in-situ and to trigger crosslinking via ionic complexation of identical charges on two or more ionomer chains, in the presence of water and a divalent metaloxide. U.S. Patent 6,180,040 teaches the formation of an ionomer via photopolymerization using energy curable compositions of ionic monomers such as metal acrylic carboxylates copolymerized with polybutadiene resins. These compounds form ionomers upon polymerization and are bridged over a metal complex in a second (vulcanization) step. In neither of these examples is the bridging ion an organic polymer, either preexisting or formed by in-situ polymerization. Fundamentally,

the ionic complexation routes illustrated in the above references yield materials with regular, repeating crosslink patterns and compact ionic structures.

Ionomers that crosslink only over ethylenically unsaturated moieties (i.e., do not employ either bridging ions or acid/base combinations of separate, oppositely charged ionomers or monomers) are commonly used in coatings and photoresists. These compounds are usually water soluble polymers carrying ethylenically unsaturated groups in or grafted onto the polymer chain of the single-chain ionomer. Examples of these polymer types are neutralized acrylics described in U.S. Patent 4,275,142; styrene-maleic anhydride described in U.S. Patent 3,825,430 and 4,401,793; and polyester, or urethane polymer salts described in U.S. Patents 6,207,346 and 5,554,712. Since no use is made of the ionic groups to create additional crosslinks, cured films made from these ionomers are often structurally weakened by the existence of ionic charges, as these sites retain moisture, which plasticizes the cured final polymer (e.g., they show lower rub resistance to water compared to uncharged polymers).

U.S. Patents 4,745,138; 5,868,605 and 6,099,415 disclose the use of chemically similar but non-neutralized resins in energy curing compositions. However, these patents do not teach the formation of blends with resins, oligomers, or monomers that could potentially neutralize an energy curable resin in a way that might crosslink the polymer. In addition, the viscosity of these non-neutralized resins is typically high. To bring them to application viscosity they must be diluted with either a large amount of a low viscosity reactive monomer or with a solvent.

SUMMARY OF THE INVENTION

The invention is the formation and use of water soluble energy curable ionomers that form a stereo crosslinked network upon curing over ethylenically unsaturated and polyionic sites. As a result, energy curable stereo-crosslinkable ionomers are produced that deliver a low viscosity liquid and create a cured solid film having superior mechanical and solvent resistance properties along with good adhesion to difficult substrates. The materials also resist cracking and flaking, and offer improved gloss and rub resistance, and enhanced coverage when compared to existing materials used in formulating paints, inks, and coatings.

DETAILED DESCRIPTION

14

The present invention shows how in-situ photopolymerization can be used to generate the opposing charge type of polymer from low-viscosity liquid monomer/oligomer blends that have utility in the manufacture of coated and printed materials. The structures formed in the present invention are random, largely amorphous, three-dimensional networks of opposing charge polymers with control over the rigidity of the crosslink. The invention employs lower molecular weight oligomeric resins and water as solvent to reduce viscosity and accelerate cure at zero volatile organic content (VOC). Cure occurs in the presence of the water, and the dissolved water is allowed to concurrently dry without application of additional energy to give cured structures that are surprisingly not sensitive to water. The oligomeric resins are neutralized with ethylenically unsaturated polyamines to form water-soluble resin salts. In some instances these salts are liquids (low melting solids), but more generally they require a water content above 10 % to be fluid. In most instances, in order to provide useful viscosity, the water content will be above 30%.

In water based energy curable compositions, viscosity reducing monomeric compounds, typically employed in energy curable compositions are replaced with water. There are two fundamentally different technologies used in this field. One derives from ethylenically unsaturated water based emulsions, which are dried before curing. The other is based on partially soluble energy curable material where the curing reaction is carried out in solution and does not necessarily include a drying step before cure. The precursors employed in the present invention are water soluble at least partially water-soluble, a state that is obtained from the use of truly water soluble monomers and oligomers in admixture with the required ionic materials.

Ethylenically Unsaturated Resin

The water soluble ethyleneically unsaturated oligomeric or polymeric resin may have acid functional groups (e.g. carboxylic acid groups) which are partially or totally neutralized with a base (e.g., an amine) to form a water soluble resin salt. Alternatively, the resin may have basic functional groups (e.g. amino groups) which are partially or totally neutralized with an acid (e.g. a carboxylic acid) to form a water soluble resin salt. A preferred embodiment is a neutralization product, where a water soluble, acrylated, resin salt is formed from an ethylenically unsaturated energy curable resin containing acrylic groups, methacrylic groups or a combination thereof; and carboxylic acid

functional groups, neutralized by a base. While a more preferred resin salt product is a neutralization product prepared from an ethylenically unsaturated amine and a polyanionic resin. Suitable examples of a polyanionic resin are polyacrylic or styrene-maleic anhydride copolymers, containing carboxyl groups, and having an acid value of at least 80 (mg KOH per 100 g polymer). Commercially available examples of such resin are Carboset GA-1167 from BF Goodrich; Joncryl 690 from SC Johnson; and SMA 1000 from ELF Atochem. It is preferred that the polyanionic resin be partially esterified via a polymer analog reaction to tailor the final properties of the compound and the final product of compositions containing the compound (propanol, isopropanol, stearic alcohol, polypropylene glycol) but that such resin have the same acid number of 80. A preferred modification uses an ethylenically unsaturated alcohol to form an ethylenically unsaturated polyanionic resin containing at least two such functions per molecule. The resin is then neutralized to a pH of at least 5.5 with an ethylenically unsaturated amine or a mixture such amine and ammonia or other caustic component. If there is no ethylenically unsaturated content in the polyanionic resin, then the tertiary amine should be ethylenically unsaturated. If the polyanionic resin is ethylenically unsaturated, then the tertiary amine may be saturated provided that it contains at least two amine groups per molecule. However, it is most preferred that both components of the resin be ethylenically unsaturated.

A particularly preferred energy curable resin is a styrene/maleic anhydride copolymer partially esterified with a hydroxy alkyl acrylate or methacrylate. The hydroxy alkyl acrylate or methacrylate preferably of such resin is preferably hydroxy butyl acrylate or methacrylate. A partially esterified styrene/maleic anhydride copolymer may be neutralized without further modification or it may be further partially esterified with an alkanol such as butanol, propanol, ethanol and the like.

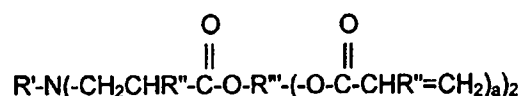
The acidic or carboxylic acid (anhydride) groups of the energy curable resin are partially or totally neutralized to provide a resin having the desired range of water solubility while retaining complete miscibility with other water soluble resins.

The resins used to form the compositions of the present invention while having an acid number of at least 80 have a molecular weight between 1,000 and 25,000; and more preferably between 1,000 and 10,000; and most preferably between 1,000 and 5,000.

16

Any basic compound (e.g., alkali metal hydroxides such as calcium hydroxide, potassium, hydroxide and sodium hydroxide, ammonia, amines, etc.) may be used to neutralize the acidic groups of the resin. Preferred are ammonia, amines or combinations thereof. Even more preferred is amines, while more particularly preferred is an ethylenically unsaturated tertiary amine. By employing ethylenically unsaturated tertiary amines as the neutralizing agent, the acidic groups on the energy curable resin are totally neutralized to form a water soluble resin having additional polymerizable ethylenic groups. The use of ethylenically unsaturated tertiary amines as the neutralizing agent further allows the acid groups on the resin to be totally neutralized which aids in the formation of the stereo cross-linkable water soluble ionomers of the present invention.

The ethylenically unsaturated tertiary amine neutralizing agent of the present invention has the formula:



wherein R' is a short chain hydrocarbon group; R'' is H or a methyl group; and R''' is selected from the group consisting of C₁-C₂₀ alkyl, C₁-C₂₀ aralkyl, C₁-C₂₀ alkyl substituted aralkyl and C₁-C₂₀ oxyalkylated derivatives; and a is 1, 2, or 3.

Preferably the ethylenically unsaturated tertiary amine is a Michael Addition product of a primary or secondary amine (e.g., an alkyl amine) and two acrylic esters, wherein each of the acrylic esters contains two or more acrylate or methacrylate groups (e.g., wherein the acrylic ester is an acrylate ester or methacrylate ester of an alkane diol, a polyether diol, a glycol, a glycerol). The primary or secondary amine may be selected, for example, from ethyl amines, ethanol amines, diethanol amines and hexamethylene imines and combinations thereof. The acrylic ester may be selected from hexanediol diacrylate, dipropylene glycol diacrylate (DPGDA), tripropylene glycol diacrylate (TPGDA), trimethylolpropane triacrylate (TMPTA), pentaerythritol triacrylate (PETA) and combinations thereof. Examples of commercially available acrylic functional amines are Laromer 8996 (available from BASF); Ebecryl 7100 (available from UCB); Suncure 175 (available from Reichhold); and Ebecryl P-104 (available from UCB).

Stereo Cross-Linkable Ionomer

It should be noted that the monomeric/oligomeric nature of the ionomer components results in a random distribution of the cationic and anionic charges within the ionomer network (see Figure 1). This differs from existing preformed polymeric ionomer pairs in which one of the pair of polymers contains cationic charges while the other contains anionic charges (see Figure 2). In the present invention, stereo cross-linkable is defined as the ability of the oligomeric ionomers to randomly polymerize in-situ via two different mechanisms. The first mechanism being a covalent free radical photopolymerization of the resin. The second mechanism being an ionic cross-link between the acidic and basic functional groups of the resin in multiple dimensions at the same time, to form a highly crosslinked polymeric network of infinite molecular mass. For example, the ethylenically unsaturated tertiary amine neutralizing agent provides the counter ion for the acidic ethylenically unsaturated resin, which allows the ionomer formed, to stereo polymerize during photoreaction (via energy curing) and form an additional cross-linked network over the ethylenically unsaturated groups as well as over the ionic structure of the resin. Therefore, unlike other water based energy curable resin technology, where the resistance properties to be imparted by the cured resin composition depend on, and are a function of, the evaporation of the base (e.g. ammonia), for example, which shifts the acid base equilibrium in the post cure composition, here for example, the ethylenically unsaturated tertiary amine neutralizing base, and neutralized resin form an additional cross-linked network instantly on both sides of the ionomer.

Upon irradiation, either or both of the ions formed from the two oligomeric material formations increase in molecular weight by addition polymerization. The use of (meth)acrylic functional, carboxylic polymeric, and tertiary amine oligomeric resins in an energy curing mechanism results in the formation of a crosslinked ionomer with a ladder structure via the formation of covalent bonds as well as opposing ionic bonds. It is essential that one oligomeric material polymerizes but it is preferred that they both polymerize. After polymerization, a highly crosslinked structure is formed showing exceptional properties over conventional and "water-compatible" energy curable materials existing in the prior art. The use of the present composition in energy curable leads to improved cure, adhesion, hardness, mechanical and solvent resistance properties.

The water solubility of the polymeric resin salt of the present invention makes it especially suitable for water based energy curing processes while the organic acid/base

crosslinking works against the tendency of ionic polymers to become mechanically weaker upon absorption of water. The control of the molecular weight between acidic and basic sites on separate oligomers allows for the formation of highly crosslinked materials which show much less brittle failure than expected for crosslinked ionomers.

For the synthesis of the resin salt three processes can be used. The first process starts from a solid carboxylic resin, which will be diluted in tertiary acrylic amine and optionally, water. The second process starts with the polymer analog reaction of alcohols with the styrene maleic anhydride resin in a solvent. In a solvent stripping step, the resin will be neutralized and diluted with tertiary acrylic amine. In the third process the solvent of the previous described ester modified resin will be azeotropically distilled with water and partially neutralized with ammonia. Afterward the resin is further neutralized with acrylated amine at 60°C

Example 1 (Comparative)

SMA-1000 (40 grams, available from Atofina) solid is slurried into water (50 grams) and neutralized to pH 6.5 with concentrated NH_4OH (available from Fisher Scientific). The resulting solution was 43 % in solids content and had a 31 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

Example 2

SMA-1000 (40 grams, available from Atofina) solids is slurried into water (50 grams) and neutralized to pH 6.5 with di(2-hydroxyethyl)methyl amine (15 grams, available from Aldrich) with heating to 60 °C for 12 hours. The resulting solution was 50 % solids in content and had a 30 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

Example 3

SMA-1000 (35 grams, available from Atofina) solids is slurried into water (50 grams) and neutralized to pH 4.5 with concentrated NH_4OH (5 grams, available from Fisher Scientific) followed by addition of acrylated amine 16-101 (10 grams, available from Reichhold) and heating to 60 °C for 4 hours. After cooling, the resulting solution was corrected to pH 6.5 with additional concentrated ammonia and measured to be 48 % in solids content and had a 48 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

19

Example 4 (Comparative)

As described in US Patent 6,559,222, polymeric resin salt, styrene maleic anhydride copolymer (165 grams) having an acid number of 480 and an average molecular weight of 1000 were added together under agitation to methyl isobutyl ketone (MIBK, 120 grams). The two materials were then heated to approximately 95-110 degrees C. over 1 to 2 hours under a nitrogen blanket. Next, N, N-dimethylbenzyl amine (0.8 grams) and a monofunctional alcohol (18 grams) such as n-propanol, ethanol or octadecanol were then added to form a polymeric mixture having an acid number between 200 to 210. The nitrogen blanket was then removed and 4-methoxyphenol (0.12 grams) and N, N-dimethylbenzylamine (0.36 grams) were added. Over a period of time, for example 60 to 90 minutes, a hydroxy-functional acrylate such as 4-hydroxybutyl acrylate (55.80 grams) or 2-hydroxy-ethyl acrylate was then added until the acid number of the polymeric mixture is between 130 to 140. The polymeric mixture was then distilled and 4-methoxyphenol (0.12 grams) was added along with ammonium hydroxide (27.90 grams) and deionized water (327.8 grams). The mixture was then heated, for example to 99 degrees C. The MIBK and water were then removed by distillation. When all of the MIBK had been removed, the water is returned to the mixture as a water/ammonia distillate. This material was prepared as a 37 % in solids content in water and neutralized to pH 6.5 with ammonia yielding 23 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

Example 5

To the resin salt solution (108 grams) as prepared in Example 4 at pH 4.5 (containing 37% resin solids in water prior to the final addition of neutralizing base described in US Patent 6,559,222) at 60 °C was added di(2-hydroxyethyl)methyl amine (10 grams, Aldrich). After cooling, the resulting solution had a pH of 6.5 and was 43 % in solids content and 20 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

Example 6

To resin salt solution (100 grams) as prepared in Example 4 at pH 4.5 (containing 37% resin solids in water prior to the final addition of neutralizing base described in US Patent 6,559,222) at 60 °C was added acrylated amine 16-101 (15 grams, Relchhold) over four hours. Upon cooling, the resulting solution was corrected to pH 6.5 with concentrated ammonia and measured to be 48 % in solids content and had a 37 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

20

Example 7 (Comparative)

Laromer 8765 (20 grams, available from BASF Corporation, Mount Olive, NJ) was added to the resin salt solution (25 grams) as prepared in Example 1. Irgacure 2959 (1.5 grams, available from Ciba) was then added to this solution. To complete the solution for coating, water (3 grams) and TegoRad 2200N (0.5 grams available from TegoRad Corporation) were added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of coating solution was 0.35 Pa.s. at 25 °C.

Example 8

Laromer 8765 (17.5 grams, available from BASF) followed by Irgacure 2959 (1.5 grams, available from Ciba) were added with stirring to resin salt solution in water (27 grams) as prepared in Example 2. water (3.5 grams) and TegoRad 2200N (0.5 grams available from TegoRad Corporation) were then added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of coating solution was 0.38 Pa.s. at 25 °C.

Example 9

Laromer 8765 (17.5 grams, BASF) followed by Irgacure 2959 (1.5 grams, available from Ciba) were added with stirring to resin salt solution in water (30.5 grams) as prepared in Example 3. TegoRad 2200N (0.5 grams available from TegoRad Corporation) was then added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of coating solution was 0.52 Pa.s. at 25 °C.

Example 10 (Comparative)

Laromer 8765 (17.5 grams, BASF) followed by Irgacure 2959 (1.5 grams, available from Ciba) were added with stirring to resin salt solution in water (30.5 grams) as prepared in Example 4. TegoRad 2200N (0.5 grams, available from TegoRad Corporation) was then added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of the coating solution was 0.23 Pa.s. at 25 °C.

Example 11

Laromer 8765 (17.5 grams, available from BASF) followed by Irgacure 2959 (1.5 grams, available from Ciba) were added with stirring to resin salt solution in water (30.5 grams) as prepared in Example 5. TegoRad 2200N (0.5 grams, available from TegoRad Corporation) was then added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of the coating solution was 0.28 Pa.s. at 25 °C.

Example 12

Laromer 8765 (18.0 grams, available from BASF) followed by Irgacure 2959 (1.5 grams, available from Ciba) were added with stirring to resin salt solution in water (30 grams) as prepared in Example 6. TegoRad 2200N (0.5 grams, available from TegoRad Corporation) was then added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of the coating solution was 0.25 Pa.s. at 25 °C.

Example 13

The coating solutions described in Examples 7 to 12 were applied by #3 and #5 wire-wound rods to Uncoated Leneta N2A charts (Leneta is a product and trademark of The Leneta Company, 15 Whitney Rd, Mahwah, NJ). Immediately following coating, the wet film was cured by passing under 650 mJ/cm² of UV light (two medium pressure Hg lamps at 300 W/in each, 200 fpm on an RPC Industries processor) in air. The resulting cured surfaces were conditioned at 75 °F and 48 % Relative Humidity for one day and the following measurements taken. Gloss was measured at 60-degree angle using a type DIN Geproft 4501 meter from BYK Gardner parallel to the coating direction. The rub resistance (an methyl ethyl ketone (MEK) rub and water rub test) was determined by wetting the cured coating surface and employing light finger pressure to rub the coating off as detected by the exposure of the underlying ink. The number of complete back-and-forth cycles required were recorded. Coating adhesion was measured by taking a convenient length of 610 tape (available from 3M Co., St. Paul, MN), laminating the tape to the cured surface under finger pressure, then lifting the tape from the surface in one rapid motion at right angle to the coated surface. The adhesion was rated a pass when the coating remained completely intact and adhered to the substrate following tape peel. The coating weight was determined gravimetrically by the difference in weight between a 10 cm X 10 cm piece cut from the coated area and an

identical size piece cut from a similar area of uncoated stock. Each measurement reported in Table 1 below is normalized to the same dry coating weight (4 g/m²).

Table 1

<u>Example</u>	<u>Gloss</u>	<u>MEK Rub</u>	<u>Water Rub</u>	<u>Adhesion</u>
7 (comparative)	65	6	5	fail
10 (comparative)	89	12	18	pass
11	91	12	16	pass
12	95	35	20	pass

Those skilled in the art having the benefit of the teachings of the present invention as hereinabove set forth, can effect numerous modifications thereto. These modifications are to be construed as being encompassed within the scope of the present invention as set forth in the appended claims.

CLAIMS

23

What we claim is:

1. An aqueous, energy curable, homogenous, composition comprising the neutralization product of
 - (a) an ethylenically unsaturated acidic resin containing carboxylic acid, acrylic functional groups, methacrylic functional groups or a combination thereof, and
 - (b) an ammonia, amine, alkali metal hydroxide or a combination thereof; in
 - (c) water;where upon curing with an actinic radiation source, a stereo-crosslinked ionomer forms offering the composition an increased cross-linked density.
2. The composition of Claim 1 wherein the, ethylenically unsaturated resin is a styrene/maleic anhydride copolymer, partially esterified with a hydroxy alkyl acrylate or methacrylate functional group.
3. The composition of Claim 2 wherein the partially esterified styrene/maleic anhydride copolymer is further esterified with an alcohol group.
4. The composition of Claim 1 wherein the neutralizing agent is ammonia, ethylenically unsaturated amine, or a combination thereof.
5. The composition of Claim 4 wherein the neutralizing agent is ammonia, ethylenically unsaturated tertiary amine, or a combination thereof
6. The composition according to Claim 1, wherein the ethylenically unsaturated resin has an acid number of at least 80 and a weight average molecular weight between 1,000 and 50,000.
7. The composition according to Claim 6, wherein the ethylenically unsaturated resin has an acid number of at least 80 and a weight average molecular weight between 1,000 and 25,000.

- 24
8. The composition according to Claim 7, wherein the ethylenically unsaturated resin has an acid number of at least 80 and a weight average molecular weight between 1,000 and 10,000.
9. An aqueous, energy curable, homogenous, composition comprising:
the neutralization product of
(d) an ethylenically unsaturated basic resin containing amine, acrylic functional groups, methacrylic functional groups or a combination thereof and
(e) an acid; in
(f) water,
where upon curing with an actinic radiation source, a stereo-crosslinked ionomer forms offering the composition an increased cross-linked density.
10. An aqueous, energy curable, homogenous, composition comprising:
the neutralization product of
(a) an ethylenically unsaturated styrene/maleic anhydride copolymer acidic resin, partially esterified with a hydroxy alkyl acrylate, further esterified by an alcohol, containing carboxylic acidic functional groups; and
(b) an ammonia, ethylenically unsaturated tertiary amine, or a combination thereof; in
(c) water,
where upon curing with an actinic radiation source, a stereo-crosslinked ionomer forms offering the composition an increased cross-linked density.
11. A method for preparing an energy curable coating comprising employing the composition of Claim 1.
12. A method for preparing an energy curable coating comprising employing the composition of Claim 9.
13. A method for preparing an energy curable coating comprising employing the composition of Claim 10.
14. An energy curable coating comprising the composition of Claim 1.

15. An energy curable coating comprising the composition of Claim 9.

16. An energy curable coating comprising the composition of Claim 10.

25

COMPOSICIONES DE IONÓMEROS ESTÉREO-RETICULABLES CURABLES POR
ENERGÍA SOLUBLES EN AGUA

Campo de la Invención

5 Esta invención se refiere a la síntesis y uso de
ionómeros estéreo reticulables curables por energía solubles
en agua, usados en la manufactura de materiales recubiertos e
impresos.

Antecedentes de la Invención

10 Los ionómeros son compuestos poliméricos que llevan
cargas electrónicas dentro de la cadena polimérica. Los
ionómeros acumulan propiedades excepcionales como dureza y
resistencia a solvente cuando dos o más cadenas poliméricas
15 crean una estructura tipo escalera por la formación de sal.
La formación de complejos iónicos de cargas idénticas u
opuestas (por ejemplo, puenteadas por un contador iónico de
valencia superior) (por ejemplo, neutralización ácido/base de
amina funcional con especies poliméricas carboxilo-
20 funcionales) en las cadenas poliméricas, resulta en
reticulaciones adicionales, el número por unidad de volumen
el cual determina la resistencia mecánica del sólido
resultante. Las rutas de formaciones de complejos iónicos más
comúnmente usadas típicamente proceden a través del uso de un
25 complejo más estable del contador iónico de puente con un

material volátil (por ejemplo, amoníaco), para permitir mezclado en el estado líquido seguido por reticulación posterior después del secado a un sólido. La neutralización ácido/base de dos ionómeros cargados contrariamente crea
5 reticulación, por el contrario, es ampliamente impráctica debido a la imposibilidad de mezclar estos materiales para generar cualquier otro distinto a un sólido reticulado insoluble.

El procedimiento de fotopolimerización para
10 reticulación de ionómeros no es nuevo. Las Patentes Estadounidenses 6,281,271 y 6,017,982, describen la curación por energía de iones etilénicamente insaturados para acumular peso molecular covalente in situ y activar la reticulación vía formación de complejos iónicos de cargas idénticas en dos
15 o más cadenas de ionómeros, en la presencia de agua y un metalóxido divalente. La Patente Estadounidense 6,180,040, muestra la formación de un ionómero vía fotopolimerización usando composiciones curables por energía de monómeros iónicos, tales como carboxilatos acrílicos metálicos
20 copolimerizados con resinas de polibutadieno. Estos compuestos forman ionómeros después de la polimerización y son puenteados sobre un complejo metálico en una segunda etapa (vulcanización). En ninguno de estos ejemplos el ión de puente es un polímero orgánico, ya sea preexistente o formado
25 por polimerización in situ. Fundamentalmente, las rutas

formadoras de complejos iónicos ilustradas en las referencias anteriores, proporciona materiales con patrones de reticulación, repetición, regulares y estructuras iónicas compactas.

5 Los ionómeros que se reticulan solamente sobre porciones etilénicamente insaturadas (es decir, no emplean ya sea iones de puentes o combinaciones ácido/base de ionómeros o monómeros opuestamente cargados, separados), son comúnmente usados en recubrimientos y fotorresistencias. Estos
10 compuestos son usualmente polímeros solubles en agua que llevan grupos etilénicamente insaturados en o injertados sobre la cadena polimérica del ionómero de cadena única. Ejemplos de estos tipos de polímeros son acrílicos neutralizados descritos en la Patente Estadounidense
15 4,275,142; anhídridos estireno-maleicos descritos en la Patente Estadounidense 3,825,430 y 4,401,793; y poliéster, o sales poliméricas de uretano descritas en las Patentes Estadounidenses 6,207,346 y 5,554,712. Puesto que no se hace uso de los grupos iónicos para crear reticulaciones
20 adicionales, las películas curadas elaboradas de estos ionómeros son a menudo estructuralmente debilitadas por la existencia de cargas iónicas, conforme estos sitios retienen humedad, los cuales plastifican el polímero final curado (por ejemplo, muestran resistencia inferior a la fricción al agua
25 comparada con polímeros no cargados).

Las Patentes Estadounidenses 4,745,138; 5,868,605 y 6,099,415, describen el uso de resinas químicamente similares pero no neutralizadas en composiciones de curación por energía. Sin embargo, estas patentes no muestran la formación de mezclas con resinas, oligómeros o monómeros que podrían potencialmente neutralizar una resina curable por energía en una forma que podría reticular el polímero. Además, la viscosidad de estas resinas no neutralizadas es típicamente elevada. Para llevarlas a una viscosidad de aplicación, deben ser diluidas con ya sea una cantidad mayor de un monómero reactivo de viscosidad inferior o con un solvente.

Sumario de la Invención

La invención es la formación y el uso de ionómeros curables por energía solubles en agua que forman una red estero reticulada después de la curación sobre sitios etilénicamente insaturados y poliiónicos. Como un resultado, los ionómeros estéreo-reticulables curables por energía son producidos, que suministran un líquido de baja viscosidad y crean una película sólida curada que tiene propiedades de resistencia al solvente y mecánicas superiores, junto con buena adhesión a sustratos difíciles. Los materiales pueden también resistir el craqueo y descamación, y ofrecer brillo mejorado y resistencia a la fricción, y cobertura mejorada cuando se compara con materiales existentes usados en

formulación de pinturas, tintes y recubrimientos.

Descripción Detallada de la Invención

La presente invención muestra como puede ser usada la fotopolimerización in situ para generar el tipo de carga opuesta de polímero de mezclas de monómero/oligómero líquidas de baja viscosidad, que tienen utilidad en la manufactura de materiales recubiertos e impresos. Las estructuras formadas en la presente invención son redes tridimensionales, grandemente amorfas, aleatorias, de polímeros de carga opuesta con control sobre la rigidez de la reticulación. La invención emplea resinas oligoméricas de peso molecular inferior y agua como solvente para reducir la viscosidad y acelerar la curación a contenido orgánico volátil cero (COV).

La curación ocurre en la presencia del agua, y el agua disuelta se deja concurrentemente secar sin aplicación de energía adicional para dar estructuras curadas que son sorprendentemente no sensibles al agua. Las resinas oligoméricas son neutralizadas con poliaminas etilénicamente insaturadas para formar sales de resinas solubles en agua. En algunos casos, estas sales son líquidas (sólidos de bajo punto de fusión), pero más generalmente, requieren un contenido de agua arriba del 10% para ser fluidas. En muchos casos, para proporcionar viscosidad útil, el contenido de agua estará por arriba del 30%.

En composiciones curables por energía a base de agua, los compuestos monoméricos que reducen la viscosidad típicamente empleados en composiciones curables por energía, son reemplazados con agua. Existen dos tecnologías
5 fundamentalmente diferentes usadas en este campo. Una se deriva de emulsiones a base de agua etilénicamente insaturadas, las cuales se secan antes de la curación. La otra se basa en material curable por energía parcialmente soluble, en donde la reacción de curación se lleva a cabo en
10 solución y no necesariamente incluye una etapa de secado antes de la cura. Los precursores empleados en la presente invención son solubles en agua, al menos parcialmente solubles en agua, un estado que se obtiene a partir del uso de monómeros realmente solubles en agua y oligómeros en
15 mezcla con los materiales iónicos requeridos.

Resina Etilénicamente Insaturada

La resina oligomérica o polimérica etilénicamente insaturada soluble en agua, puede tener grupos funcionales
20 ácidos (por ejemplo, grupos de ácido carboxílico), los cuales son parcialmente o totalmente neutralizados con una base (por ejemplo, una amina), para formar una sal de resina soluble en agua. Alternativamente, la resina puede tener grupos funcionales básicos (por ejemplo, grupos amino), los cuales
25 son parcialmente o totalmente neutralizados con un ácido (por

ejemplo, un ácido carboxílico), para formar una sal de resina soluble en agua. Una modalidad preferida es un producto de neutralización, en donde una sal de resina acrilada, soluble en agua, se forma a partir de una resina curable por energía etilénicamente insaturada que contiene grupos acrílicos, grupos metacrílicos o una combinación de los mismos; y grupos funcionales de ácido carboxílico, neutralizados por base. Mientras, un producto de sal de resina más preferido es un producto de neutralización preparado a partir de una amina etilénicamente insaturada y una resina polianiónica. Ejemplos adecuados de una resina polianiónica son copolímeros de anhídrido estireno-maleico o poliacrílico, que contienen grupos carboxilo y que tienen un valor ácido de al menos 80 (mg KOH por 100 g de polímero). Ejemplos comercialmente disponibles de tales resinas son Carboset GA-1167 de BF Goodrich; Joncryl 690 de SC Johnson; y SMA 1000 de ELF Atochem. Se prefiere que la resina polianiónica sea parcialmente esterificada vía una reacción análoga de polímero para ajustar las propiedades finales del compuesto y el producto final de las composiciones que contienen el compuesto (propanol, isopropanol, alcohol esteárico, propilenglicol), pero que tales resinas tengan el mismo número ácido de 80. Una modificación preferida usa un alcohol etilénicamente insaturado para formar una resina polianiónica etilénicamente insaturada que contiene al menos dos de tales

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funciones por molécula. La resina es entonces neutralizada a un pH de al menos 5.5 con una amina etilénicamente insaturada o una mezcla de tal amina y amoníaco u otro componente cáustico. Si no existe contenido etilénicamente insaturado de la resina polianiónica, entonces la amina terciaria debe ser etilénicamente insaturada. Si la resina polianiónica es etilénicamente insaturada, entonces la amina terciaria puede ser saturada siempre que contenga al menos, dos grupos de amina por molécula. Sin embargo, es más preferido que ambos componentes de la resina sean etilénicamente insaturados.

Una resina curable por energía particularmente preferida es un copolímero de anhídrido estireno/maleico parcialmente esterificado con un hidroxialquilacrilato o metacrilato. El hidroxialquilacrilato o metacrilato preferiblemente de tal resina, es preferiblemente hidroxibutilacrilato o metacrilato. Un copolímero de anhídrido estireno/maleico parcialmente esterificado, puede ser neutralizado sin modificación adicional o puede ser además, parcialmente esterificado con un alcohol tal como butanol, propanol, etanol y similares.

Los grupos de ácido carboxílico (anhídridos) o ácidos de la resina curable por energía, son parcialmente o totalmente neutralizados para proporcionar una resina que tiene el intervalo deseado de solubilidad en agua, mientras retiene completa miscibilidad con otras resinas solubles en

agua.

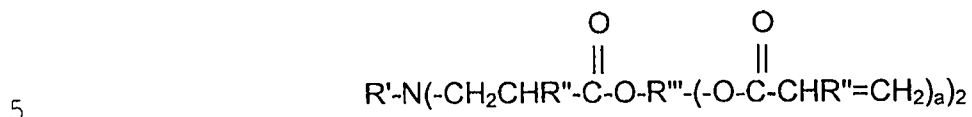
Las resinas usadas para formar las composiciones de la presente invención mientras tienen un número ácido de al menos 80, tienen un peso molecular entre 1,000 y 25,000; y
5 más preferiblemente entre 1,000 y 10,000; y mayormente de manera preferible entre 1,000 y 5,000.

Agente de Neutralización

Cualquier compuesto básico (por ejemplo, hidróxidos
10 de metal álcali tales como hidróxido de calcio, hidróxido de potasio e hidróxido de sodio, amoníaco, aminas, etc.), puede ser usado para neutralizar los grupos acídicos de la resina. Preferidos son amoníaco, aminas o combinaciones de los mismos. Aún más preferidas son aminas, mientras más
15 particularmente preferidas son aminas terciarias etilénicamente insaturadas. Empleando aminas terciarias etilénicamente insaturadas como el agente neutralizante, los grupos acídicos en la resina curable por energía son totalmente neutralizados para formar una resina soluble en
20 agua que tiene grupos etilénicos polimerizables adicionales. El uso de aminas terciarias etilénicamente insaturadas como el agente neutralizante, además permite a los grupos ácidos en la resina ser totalmente neutralizados, lo cual ayuda en la formación de los ionómeros solubles en agua estereo-
25 reticulables de la presente invención.

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El agente neutralizante de amina terciaria etilénicamente insaturado de la presente invención, tiene la fórmula:



en donde R' es un grupo hidrocarburo de cadena corta; R'' es H o un grupo metilo; y R''' se selecciona del grupo que consiste de alquilo C₁-C₂₀, aralquilo C₁-C₂₀, aralquilo sustituido con alquilo C₁-C₂₀ y derivados oxialquilados C₁-C₂₀; y a es 1, 2 ó 3.

Preferiblemente, la amina terciaria etilénicamente insaturada es un producto de Adición Michael de una amina primaria o secundaria (por ejemplo, una alquilamina) y dos ésteres acrílicos, en donde cada uno de los ésteres acrílicos contiene dos o más grupos acrilatos o metacrilatos (por ejemplo, en donde el éster acrílico es un éster acrilato o éster metacrilato de un alcandiol, un poliéter diol, un glicol, un glicerol). La amina primaria o secundaria puede ser seleccionada por ejemplo, de etilaminas, etanolaminas, dietanolaminas y hexametileniminas y combinaciones de las mismas. El éster acrílico puede ser seleccionado de diacrilato de hexandiol, diacrilato de dipropilenglicol (DPGDA), diacrilato de tripropilenglicol, (TPGDA), triacrilato de trimetilolpropano (TMPTA), triacrilato de pentaeritritol (PETA) y combinaciones de los mismos. Ejemplos

de aminos funcionales acrílicas comercialmente disponibles son Laromer 8996 (disponible de BASF); Ebecryl 7100 (disponible de UCB); Suncure 175 (disponible de Reichhold); y Ebecryl P-104 (disponible de UCB).

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Ionómero Estéreo Reticulable

Se debe notar que la naturaleza monomérica/oligomérica de los componentes ionómeros resulta en una distribución aleatoria de las cargas catiónicas y aniónicas dentro de la red de ionómero (véase Figura 1). Esto difiere de los pares de ionómeros poliméricos preformados existentes, en los cuales uno de los pares de polímeros contiene cargas catiónicas mientras el otro contiene cargas aniónicas (véase Figura 2). En la presente invención, estéreo reticulable se define como la capacidad de los ionómeros oligoméricos para polimerizar aleatoriamente in situ vía dos mecanismos diferentes. El primer mecanismo es una fotopolimerización radical libre covalente de la resina. El segundo mecanismo es una reticulación iónica entre los grupos funcionales ácidos y básicos de la resina en dimensiones múltiples al mismo tiempo, para formar una red polimérica altamente reticulada de masa molecular infinita. Por ejemplo, el agente neutralizante de amina terciaria etilénicamente insaturada, proporciona el ión contador para la resina etilénicamente insaturada ácida, lo cual permite al

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ionómero formado, estéreo polimerizarse durante la foto-reacción (vía curación por energía) y formar una red reticulada adicional sobre los grupos etilénicamente insaturados, así como también sobre la estructura iónica de la resina. Por lo tanto, a diferencia de otra tecnología de resina curable por energía a base de agua, en donde las propiedades de resistencia a ser impartidas por la composición de resina curada dependen de, y son una función de la evaporación de la base (por ejemplo amoníaco), por ejemplo, la cual cambia el equilibrio de la base ácida en la composición de cura posterior, aquí por ejemplo, la base neutralizante de amina terciaria etilénicamente insaturada y la resina neutralizada, forman una red reticulada adicional instantáneamente en ambos lados del ionómero.

Después de la irradiación, cualquiera o ambos de los iones formados de las dos formaciones de material oligomérico, incrementan en peso molecular por polimerización de adición. El uso de resinas oligoméricas de amina terciaria y carboxílicas poliméricas, (met)acrílicas funcionales en un mecanismo de curación por energía, resulta en la formación de un ionómero reticulado con una estructura de escalera vía la formación de enlaces covalentes, así como también de enlaces iónicos opuestos. Es esencial que un material oligomérico se polimerice, pero se prefiere que ambos se polimericen. Después de la polimerización, una estructura altamente

reticulada se forma mostrando propiedades excepcionales sobre materiales curables por energía "compatible con agua" y convencionales existentes en la técnica anterior. El uso de la presente composición en materiales curables por energía
5 conduce a cura mejorada, adhesión, dureza, propiedades mecánicas y de resistencia al solvente.

La solubilidad en agua de la sal de resina polimérica de la presente invención, hace especialmente adecuado los procesos de curación por energía a base de agua
10 mientras la reticulación de ácido/base orgánica funciona contra la tendencia de polímeros iónicos para volverse mecánicamente más débiles en la absorción de agua. El control del peso molecular entre sitios acídicos y básicos en oligómeros separados permite la formación de materiales
15 altamente reticulados, lo cual muestra mucho menos falla quebradiza que la esperada para ionómeros reticulados.

Para la síntesis de la sal de resina, se pueden usar tres procesos. El primer proceso parte de una resina carboxílica sólida, la cual será diluida en la amina acrílica
20 terciaria y opcionalmente agua. El segundo proceso parte con la reacción análoga polimérica de alcoholes con la resina de anhídrido estireno maleico en un solvente. En una etapa de retiro del solvente, la resina será neutralizada y diluida con amina acrílica terciaria. En el tercer proceso, el
25 solvente de la resina modificada del éster previamente

descrita, será destilada de forma azeotrópica con agua y parcialmente neutralizada con amoníaco. Posteriormente, la resina es neutralizada adicionalmente con amina acrilada a 60°C.

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Ejemplo 1 (Comparativo)

El sólido SMA-1000 (40 gramos, disponible de Atofina), es formado en suspensión en agua (50 gramos) y neutralizado a pH 6.5 con NH_4OH concentrado (disponible de Fisher Scientific). La solución resultante fue 43% de contenido de sólidos y tiene una viscosidad de 31 Pa.s. a 10 s-1 a 40°C.

Ejemplo 2

El sólido SMA-1000 (40 gramos, disponible de Atofina) es formado en suspensión en agua (50 gramos) y neutralizado a pH 6.5 con di(2-hidroxietil)metilamina (15 gramos, disponible de Aldrich) con calentamiento a 60°C por 12 horas. La solución resultante fue 50% de contenido de sólidos y tiene una viscosidad de 30 Pa.s. a 10 s-1 a 40°C.

Ejemplo 3

El sólido SMA-1000 (35 gramos, disponibles de Atofina), es formado en suspensión en agua (50 gramos) y neutralizado a pH 4.5 con NH_4OH concentrado (5 gramos,

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disponible de Fisher Scientific) seguido por la adición de amina acrilada 16-101 (10 gramos, disponible de Reichhold) y calentado a 60°C por 4 horas. Después del enfriamiento, la solución resultante fue corregida a pH 6.5 con amoníaco
5 concentrado adicional y medida para ser de 48% en contenido de sólidos y tiene una viscosidad de 48 Pa.s. a 10 s-1 a 40°C.

Ejemplo 4 (Comparativo)

Como se describe en la Patente Estadounidense
10 6,559,222, la sal de resina polimérica, el copolímero de anhídrido estireno maleico (165 gramos) que tienen un número ácido de 480 y un peso molecular promedio de 1000, fueron agregaron juntos bajo agitación a metil isobutil cetona (MIBK, 120 gramos). Los dos materiales fueron entonces
15 calentados a aproximadamente 95-110°C, durante 1 ó 2 horas bajo un manto de nitrógeno. Después, fueron agregados entonces N,N-dimetilbencilamina (0.8 gramos) y un alcohol monofuncional (18 gramos) tal como n-propanol, etanol u octadecanol, para formar una mezcla polimérica que tiene un
20 número ácido entre 200 a 210. El manto de nitrógeno entonces fue removido y se agregaron 4-metoxifenol (0.12 gramos) y N,N-dimetilbencilamina (0.36 gramos). Durante un periodo de tiempo, por ejemplo 60 a 90 minutos, un acrilato hidroxifuncional tal como 4-hidroxibutilacrilato (55.80 gramos) ó 2-
25 hidroxietil acrilato, fueron entonces agregados hasta que el

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número ácido de la mezcla polimérica está entre 130 a 140. La mezcla polimérica fue entonces destilada y se agregó 4-metoxifenol (0.12 gramos) junto con en hidróxido de amonio (27.90 gramos) y agua desionizada (327.8 gramos). La mezcla
5 fue entonces calentada, por ejemplo, a 99°C. La MIBK y el agua fueron entonces removidos por destilación. Cuando toda la MIBK ha sido removida, el agua es regresada a la mezcla como un destilado de agua/amoniaco. El material fue preparado como un 37% en contenido de sólidos en agua, y neutralizado a
10 pH 6.5 con amoniaco, proporcionando una viscosidad de 23 Pa.s. a 10 s⁻¹ a 40°C.

Ejemplo 5

A la solución de sal de resina (108 gramos) como se
15 preparó en el Ejemplo 4 a pH 4.5 (que contiene 37% de sólidos de resina en agua antes de la adición final de base neutralizante descrita en la Patente Estadounidense 6,559,222), a 60°C se agregó di(2-hidroxietil)metilamina (10 gramos, Aldrich). Después del enfriamiento, la solución
20 resultante tiene un pH de 6.5 y es de 43% en contenido de sólidos y viscosidad de 20 Pa.s. a 10 s⁻¹ a 40°C.

Ejemplo 6

A la solución de sal de resina (100 gramos) como se
25 preparó en el Ejemplo 4 a pH 4.5 (que contiene 37% de sólidos

de resina en agua antes de la adición final de base neutralizante descrita en la Patente Estadounidense 6,559,222), a 60°C se agregó amina acrilada 16-101 (15 gramos, Reichhold) durante cuatro horas. Después del
5 enriamiento, la solución resultante fue corregida a pH 6.5 con amoníaco concentrado y medida para ser 48% en contenido de sólidos y tiene una viscosidad de 37 Pa.s. a 10 s-1 a 40°C.

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Ejemplo 7 (Comparativo)

Se agregó Laromer 8765 (20 gramos, disponible de BASF Corporation, Mount Olive, NJ) a la solución de sal de resina (25 gramos), como se preparó en el Ejemplo 1. Se agregó entonces Irgacure 2959 (1.5 gramos, disponible de
15 Ciba) a esta solución. Para completar la solución para recubrimiento, se agregaron agua (3 gramos) y TegoRad 2200N (0.5 gramos disponible de TegoRad) con agitación, y la solución se dejó a un lado por 12 horas para separar el aire entrante antes del recubrimiento y curación. La viscosidad de
20 la solución de recubrimiento fue de 0.35 Pa.s. a 25°C.

Ejemplo 8

Se agregaron Laromer 8765 (17.5 gramos, disponible de BASF), seguido por Irgacure 2959 (1.5 gramos, disponible
25 de Ciba) con agitación a la solución de sal de resina en agua

(27 gramos) como se preparó en el Ejemplo 2. Fueron agregados entonces agua (3.5 gramos) y TegoRad 2200N (0.5 gramos disponible de TegoRad Corporation) con agitación, y la solución se dejó a un lado por 12 horas para separar el aire entrante antes del recubrimiento y curación. La viscosidad de la solución de recubrimiento fue de 0.38 Pa.s. a 25°C.

Ejemplo 9

Se agregaron Laromer 8765 (17.5 gramos, BASF), seguido por Irgacure 2959 (1.5 gramos, disponible de Ciba) con agitación a la solución de sal de resina en agua (30.5 gramos) como se preparó en el Ejemplo 3. Se agregó entonces TegoRad 2200N (0.5 gramos disponible de TegoRad Corporation) con agitación, y la solución se dejó a un lado por 12 horas para separar el aire entrante antes del recubrimiento y curación. La viscosidad de la solución de recubrimiento fue de 0.52 Pa.s. a 25°C.

Ejemplo 10

Se agregaron Laromer 8765 (17.5 gramos, BASF), seguido por Irgacure 2959 (1.5 gramos, disponible de Ciba) con agitación a la solución de sal de resina en agua (30.5 gramos) como se preparó en el Ejemplo 4. Se agregó entonces TegoRad 2200N (0.5 gramos disponible de TegoRad Corporation) con agitación, y la solución se dejó a un lado por 12 horas

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para separar el aire entrante antes del recubrimiento y curación. La viscosidad de la solución de recubrimiento fue de 0.23 Pa.s. a 25°C.

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Ejemplo 11

Se agregaron Laromer 8765 (17.5 gramos, disponible de BASF), seguido por Irgacure 2959 (1.5 gramos, disponible de Ciba) con agitación a la solución de sal de resina en agua (30.5 gramos) como se preparó en el Ejemplo 5. Se agregó entonces TegoRad 2200N (0.5 gramos disponible de TegoRad Corporation) con agitación, y la solución se dejó a un lado por 12 horas para separar el aire entrante antes del recubrimiento y curación. La viscosidad de la solución de recubrimiento fue de 0.28 Pa.s. a 25°C.

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Ejemplo 12

Se agregaron Laromer 8765 (17.5 gramos, disponible de BASF), seguido por Irgacure 2959 (1.5 gramos, disponible de Ciba) con agitación a la solución de sal de resina en agua (30 gramos) como se preparó en el Ejemplo 6. Se agregó entonces TegoRad 2200N (0.5 gramos disponible de TegoRad Corporation) con agitación, y la solución se dejó a un lado por 12 horas para separar el aire entrante antes del recubrimiento y curación. La viscosidad de la solución de recubrimiento fue de 0.25 Pa.s. a 25°C.

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Ejemplo 13

Las soluciones de recubrimiento descritas en los Ejemplos 7 a 12, fueron aplicadas por varillas bobinadas #3 y #5 en diagramas Leneta N2A No recubiertos (Leneta es un producto y marca comercial de The Leneta Company, 15 Whitney Rd, Mahwah, NJ.). Inmediatamente después del recubrimiento, la película húmeda fue curada pasándola bajo 650 mJ/cm² de luz UV (dos lámparas de Hg de presión media a 300 W/en cada una, 200 fpm en un procesador de RPC Industries) en aire. Las superficies curadas resultantes fueron acondicionadas a 75°F (23.8°C) y 48% de Humedad Relativa por un día y se tomaron las siguientes mediciones. El brillo fue medido a un ángulo de 60 grados usando un medidor tipo DIN Geproft 4501 de BYK Gardner paralelo a la dirección del recubrimiento. La resistencia a la fricción (una prueba de resistencia al agua y a la fricción con metiletilcetona (MEK), fue determinada humedeciendo la superficie recubierta curada y empleando presión ligera del dedo para friccionar el recubrimiento como se detecta por la exposición de la tinta subyacente. Fue registrado el número de ciclos recíprocos completos. La adhesión de recubrimiento fue medida tomando una longitud conveniente de cinta 610 (disponible de 3M Co., St. Paul, MN), laminando la cinta a la superficie curada bajo presión del dedo, después levantando la cinta de la superficie en un movimiento rápido en ángulo recto a la superficie recubierta.

La adhesión fue clasificada un paso cuando el recubrimiento permaneció completamente intacto y adherido al sustrato después del desprendimiento de la cinta. El peso del recubrimiento fue determinado gravimétricamente por la

5 diferencia en peso entre un corte de pieza de 10 cm x 10 cm a partir del área recubierta y un corte de pieza de tamaño idéntico a partir de un área similar de una pila no recubierta. Cada medición reportada en la Tabla 1 abajo, es normalizada al mismo peso del recubrimiento seco (4 gl/m^2).

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Tabla 1				
<u>Ejemplo</u>	<u>Brillo</u>	<u>Fricción</u> <u>por MEK</u>	<u>Fricción</u> <u>por Agua</u>	<u>Adhesión</u>
7 (comparativo)	65	6	5	Falla
15 10 (comparativo)	89	12	18	Paso
11	91	12	16	Paso
12	95	35	20	paso

Aquellos expertos en la técnica que tienen el

20 beneficio de las enseñanzas de la presente invención como se expone en este documento anteriormente, pueden efectuar numerosas modificaciones a ésta. Estas modificaciones son para ser construidas por estar abarcadas dentro del alcance de la presente invención como se expone en las

25 reivindicaciones adjuntas.

REIVINDICACIONES

Habiéndose descrito la invención como antecede, se reclama como propiedad lo contenido en las siguientes reivindicaciones.

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1. Una composición homogénea, curable por energía, acuosa, caracterizada porque comprende el producto de neutralización de:

10 (a) una resina ácida etilénicamente insaturada que contiene ácido carboxílico, grupos funcionales acrílicos, grupos funcionales metacrílicos o una combinación de los mismos, y

(b) un amoníaco, amina, hidróxido de metal álcali o combinación de los mismos; en

15 (c) agua;

en donde después de la curación con una fuente de radiación actínica, un ionómero estéreo-reticulable se forma ofreciendo a la composición una densidad reticulada incrementada.

20 2. La composición de conformidad con la reivindicación 1, caracterizada porque la resina etilénicamente insaturada es un copolímero de anhídrido estireno/maleico, parcialmente esterificado con un grupo funcional metacrilato o hidroxialquilacrilato.

25 3. La composición de conformidad con la

reivindicación 2, caracterizada porque el copolímero de anhídrido estireno/maleico parcialmente esterificado es además esterificado con un grupo alcohol.

4. La composición de conformidad con la
5 reivindicación 1, caracterizada porque el agente neutralizante es amoníaco, amina etilénicamente insaturada o una combinación de los mismos.

5. La composición de conformidad con la
reivindicación 4, caracterizada porque el agente
10 neutralizante es amoníaco, amina terciaria etilénicamente insaturada o una combinación de los mismos.

6. La composición de conformidad con la
reivindicación 1, caracterizada porque la resina
etilénicamente insaturada tiene un número ácido de al menos
15 80 y un peso molecular promedio en peso entre 1,000 y 50,000.

7. La composición de conformidad con la
reivindicación 6, caracterizada porque la resina
etilénicamente insaturada tiene un número ácido de al menos
80 y un peso molecular promedio en peso entre 1,000 y 25,000.

20 8. La composición de conformidad con la
reivindicación 7, caracterizada porque la resina
etilénicamente insaturada tiene un número ácido de al menos
80 y un peso molecular promedio en peso entre 1,000 y 10,000.

9. Una composición homogénea, curable por energía,
25 acuosa, caracterizada porque comprende el producto de

neutralización de:

(a) una resina básica etilénicamente insaturada que contiene amina, grupos funcionales acrílicos, grupos funcionales metacrílicos o una combinación de los mismos y

5 (b) un ácido; en

(d) agua,

en donde después de la curación con una fuente de radiación actínica, un ionómero estéreo-reticulado se forma ofreciendo a la composición una densidad reticulada
10 incrementada.

10. Una composición homogénea, curable por energía, acuosa, caracterizada porque comprende el producto de neutralización de:

(a) una resina ácida de copolímero de anhídrido
15 estireno/maleico etilénicamente insaturado, parcialmente esterificada con un hidroxialquilacrilato, además esterificado por un alcohol, que contiene grupos funcionales ácidos carboxílicos; y

(b) un amoníaco, amina terciaria etilénicamente
20 insaturada, o una combinación de los mismos; en

(c) agua,

en donde después de la curación con una fuente de radiación actínica, un ionómero estéreo-reticulado se forma ofreciendo a la composición una densidad reticulada
25 incrementada.

SO

11. Un método para preparar una composición de recubrimiento curable por energía, caracterizado porque se emplea la composición de conformidad con la reivindicación 1.

5 12. Un método para preparar una composición de recubrimiento curable por energía, caracterizado porque se emplea la composición de conformidad con la reivindicación 9.

13. Un método para preparar una composición de recubrimiento curable por energía, caracterizado porque se emplea la composición de conformidad con la reivindicación
10 10.

14. Una composición de recubrimiento curable por energía, caracterizada porque comprende la composición de conformidad con la reivindicación 1.

15 15. Una composición de recubrimiento curable por energía, caracterizada porque comprende la composición de conformidad con la reivindicación 9.

16. Una composición de recubrimiento curable por energía, caracterizada porque comprende la composición de conformidad con la reivindicación 10.

20

S

RESUMEN DE LA INVENCION

La presente invención se refiere a una composición
de recubrimiento que contiene un ionómero estéreo
5 reticulable, curable por energía, acuoso, homogéneo, y a un
método para preparar la misma.

PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

NOTIFICATION OF RECEIPT
OF SEARCH COPY

To:
SUN CHEMICAL CORPORATION
Attn. Persley, Sidney
222 Bridge Plaza South
Fort Lee, NJ 07024
UNITED STATES OF AMERICA

RECEIVED
(Article 25.1)

FEB 07 2005

Date of mailing
(day/month/year)

27/01/2005

Applicant's or agent's file reference

C-574

IMPORTANT NOTIFICATION

International application No.

POF/US2004/037222

International filing date (day/month/year)

05/11/2004

Priority date (day/month/year)

06/11/2003

Applicant

SUN CHEMICAL CORPORATION

1. Where the International Searching Authority and the receiving Office are not the same office:

The applicant is hereby notified that the search copy of the international application was received by this International Searching Authority on the date indicated below.

Where the International Searching Authority and the receiving Office are the same office:

The applicant is hereby notified that the search copy of the international application was received on the date indicated below.

31/12/2004 (date of receipt).

2. ☐ The search copy was accompanied by a nucleotide and/or amino acid sequence listing or tables related thereto in computer readable form.

3. Time limit for establishment of International search report and written opinion of the International Searching Authority

The applicant is informed that the time limit for establishing the international search report and the written opinion of the International Searching Authority is three months from the date of receipt indicated above or nine months from the priority date, whichever time limit expires later (Rules 42.1 and 43bis.1(a))

4. A copy of this notification has been sent to the International Bureau and, where the first sentence of paragraph 1 applies, to the receiving Office.

Name and mailing address of the International Searching Authority



European Patent Office, P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

ISA/EP

PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

53

To:

SUN CHEMICAL CORPORATION
Attn. Persley, Sidney
222 Bridge Plaza South
Fort Lee, NJ 07024
UNITED STATES OF AMERICA

RECEIVED
MAR 28 2005
PCT/ISA/220

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL SEARCH REPORT AND
THE WRITTEN OPINION OF THE INTERNATIONAL
SEARCHING AUTHORITY, OR THE DECLARATION

(PCT Rule 44.1)

Date of mailing (day/month/year) 24/03/2005	
Applicant's or agent's file reference C-574	FOR FURTHER ACTION See paragraphs 1 and 4 below
International application No. PCT/US2004/037222	International filing date (day/month/year) 05/11/2004
Applicant SUN CHEMICAL CORPORATION	

1. ☒ The applicant is hereby notified that the international search report and the written opinion of the International Searching Authority have been established and are transmitted herewith.

Filing of amendments and statement under Article 19:

The applicant is entitled, if he so wishes, to amend the claims of the International Application (see Rule 46):

When? The time limit for filing such amendments is normally 2 months from the date of transmittal of the International Search Report; however, for more details, see the notes on the accompanying sheet.

Where? Directly to the International Bureau of WIPO, 34 chemin des Colombettes
1211 Geneva 20, Switzerland, Facsimile No.: (41-22) 740.14.35

For more detailed instructions, see the notes on the accompanying sheet.

2. ☐ The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect and the written opinion of the International Searching Authority are transmitted herewith.

3. ☐ **With regard to the protest** against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

- ☐ the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.
☐ no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.

4. Reminders

Shortly after the expiration of **18 months** from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in Rules 90bis.1 and 90bis.3, respectively, before the completion of the technical preparations for international publication.

The applicant may submit comments on an informal basis on the written opinion of the International Searching Authority to the International Bureau. The International Bureau will send a copy of such comments to all designated Offices unless an international preliminary examination report has been or is to be established. These comments would also be made available to the public but not before the expiration of 30 months from the priority date.

Within **19 months** from the priority date, but only in respect of some designated Offices, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until **30 months** from the priority date (in some Offices even later); otherwise, the applicant must, **within 20 months** from the priority date, perform the prescribed acts for entry into the national phase before those designated Offices.

In respect of other designated Offices, the time limit of **30 months** (or later) will apply even if no demand is filed within 19 months.

See the Annex to Form PCT/IB/301 and, for details about the applicable time limits, Office by Office, see the *PCT Applicant's Guide*, Volume II, National Chapters and the WIPO Internet site.

Name and mailing address of the International Searching Authority  European Patent Office, P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Monika Schmitz
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NOTES TO FORM PCT/ISA/220

These Notes are intended to give the basic instructions concerning the filing of amendments under article 19. The Notes are based on the requirements of the Patent Cooperation Treaty, the Regulations and the Administrative Instructions under that Treaty. In case of discrepancy between these Notes and those requirements, the latter are applicable. For more detailed information, see also the PCT Applicant's Guide, a publication of WIPO.

59

In these Notes, "Article", "Rule", and "Section" refer to the provisions of the PCT, the PCT Regulations and the PCT Administrative Instructions respectively.

INSTRUCTIONS CONCERNING AMENDMENTS UNDER ARTICLE 19

The applicant has, after having received the international search report, one opportunity to amend the claims of the international application. It should however be emphasized that, since all parts of the international application (claims, description and drawings) may be amended during the international preliminary examination procedure, there is usually no need to file amendments of the claims under Article 19 except where, e.g. the applicant wants the latter to be published for the purposes of provisional protection or has another reason for amending the claims before international publication. Furthermore, it should be emphasized that provisional protection is available in some States only.

What parts of the international application may be amended?

Under Article 19, only the claims may be amended.

During the international phase, the claims may also be amended (or further amended) under Article 34 before the International Preliminary Examining Authority. The description and drawings may only be amended under Article 34 before the International Examining Authority.

Upon entry into the national phase, all parts of the international application may be amended under Article 28 or, where applicable, Article 41.

When?

Within 2 months from the date of transmittal of the international search report or 16 months from the priority date, whichever time limit expires later. It should be noted, however, that the amendments will be considered as having been received on time if they are received by the International Bureau after the expiration of the applicable time limit but before the completion of the technical preparations for international publication (Rule 46.1).

Where not to file the amendments?

The amendments may only be filed with the International Bureau and not with the receiving Office or the International Searching Authority (Rule 46.2).

Where a demand for international preliminary examination has been/is filed, see below.

How?

Either by cancelling one or more entire claims, by adding one or more new claims or by amending the text of one or more of the claims as filed.

A replacement sheet must be submitted for each sheet of the claims which, on account of an amendment or amendments, differs from the sheet originally filed.

All the claims appearing on a replacement sheet must be numbered in Arabic numerals. Where a claim is cancelled, no renumbering of the other claims is required. In all cases where claims are renumbered, they must be renumbered consecutively (Administrative Instructions, Section 205(b)).

The amendments must be made in the language in which the international application is to be published.

What documents must/may accompany the amendments?

Letter (Section 205(b)):

The amendments must be submitted with a letter.

The letter will not be published with the international application and the amended claims. It should not be confused with the "Statement under Article 19(1)" (see below, under "Statement under Article 19(1)").

The letter must be in English or French, at the choice of the applicant. However, if the language of the international application is English, the letter must be in English; if the language of the international application is French, the letter must be in French.

NOTES TO FORM PCT/ISA/220 (continued)

The letter must indicate the differences between the claims as filed and the claims as amended. It must, in particular, indicate, in connection with each claim appearing in the international application (it being understood that identical indications concerning several claims may be grouped), whether

- (i) the claim is unchanged;
- (ii) the claim is cancelled;
- (iii) the claim is new;
- (iv) the claim replaces one or more claims as filed;
- (v) the claim is the result of the division of a claim as filed.

The following examples illustrate the manner in which amendments must be explained in the accompanying letter:

1. [Where originally there were 48 claims and after amendment of some claims there are 51]:
"Claims 1 to 29, 31, 32, 34, 35, 37 to 48 replaced by amended claims bearing the same numbers; claims 30, 33 and 36 unchanged; new claims 49 to 51 added."
2. [Where originally there were 15 claims and after amendment of all claims there are 11]:
"Claims 1 to 15 replaced by amended claims 1 to 11."
3. [Where originally there were 14 claims and the amendments consist in cancelling some claims and in adding new claims]:
"Claims 1 to 6 and 14 unchanged; claims 7 to 13 cancelled; new claims 15, 16 and 17 added." or
"Claims 7 to 13 cancelled; new claims 15, 16 and 17 added; all other claims unchanged."
4. [Where various kinds of amendments are made]:
"Claims 1-10 unchanged; claims 11 to 13, 18 and 19 cancelled; claims 14, 15 and 16 replaced by amended claim 14; claim 17 subdivided into amended claims 15, 16 and 17; new claims 20 and 21 added."

"Statement under article 19(1)" (Rule 46.4)

The amendments may be accompanied by a statement explaining the amendments and indicating any impact that such amendments might have on the description and the drawings (which cannot be amended under Article 19(1)).

The statement will be published with the international application and the amended claims.

It must be in the language in which the international application is to be published.

It must be brief, not exceeding 500 words if in English or if translated into English.

It should not be confused with and does not replace the letter indicating the differences between the claims as filed and as amended. It must be filed on a separate sheet and must be identified as such by a heading, preferably by using the words "Statement under Article 19(1)."

It may not contain any disparaging comments on the international search report or the relevance of citations contained in that report. Reference to citations, relevant to a given claim, contained in the international search report may be made only in connection with an amendment of that claim.

Consequence if a demand for international preliminary examination has already been filed

If, at the time of filing any amendments under Article 19, a demand for international preliminary examination has already been submitted, the applicant must preferably, at the same time of filing the amendments with the International Bureau, also file a copy of such amendments with the International Preliminary Examining Authority (see Rule 62.2(a), first sentence).

Consequence with regard to translation of the international application for entry into the national phase

The applicant's attention is drawn to the fact that, where upon entry into the national phase, a translation of the claims as amended under Article 19 may have to be furnished to the designated/elected Offices, instead of, or in addition to, the translation of the claims as filed.

For further details on the requirements of each designated/elected Office, see Volume II of the PCT Applicant's Guide.

PATENT COOPERATION TREATY

PCT

56

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference C-574	FOR FURTHER ACTION see Form PCT/ISA/220 as well as, where applicable, item 5 below.	
International application No. PCT/US2004/037222	International filing date (day/month/year) 05/11/2004	(Earliest) Priority Date (day/month/year) 06/11/2003
Applicant SUN CHEMICAL CORPORATION		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 4 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ The international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, see Box No. I.

2. ☐ **Certain claims were found unsearchable** (See Box II).

3. ☐ **Unity of invention is lacking** (see Box III).

4. With regard to the **title**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. With regard to the **drawings**,

- a. the figure of the **drawings** to be published with the abstract is Figure No. _____

☐ as suggested by the applicant.

☐ as selected by this Authority, because the applicant failed to suggest a figure.

☐ as selected by this Authority, because this figure better characterizes the invention.

- b. ☐ none of the figures is to be published with the abstract.

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08F8/00 C08F8/44

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 7 C08F C09D

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)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 559 222 B1 (ROONEY JOHN ET AL) 6 May 2003 (2003-05-06) cited in the application the whole document -----	1-8, 10, 11, 13, 14, 16
X	US 4 745 138 A (THANAWALLA ET AL) 17 May 1988 (1988-05-17) cited in the application the whole document -----	1, 4-8, 11, 14
X	US 3 425 977 A (MARTIN SKOULTCHI ET AL) 4 February 1969 (1969-02-04) the whole document -----	1, 4-8, 11, 14
X	US 3 862 067 A (MOTIER ET AL) 21 January 1975 (1975-01-21) the whole document -----	1, 4-8, 11, 14
	-/--	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *I* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *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
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *G* document member of the same patent family

Date of the actual completion of the international search

10 March 2005

Date of mailing of the international search report

24/03/2005

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Droghetti, A

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 633 307 A (DAS ET AL) 27 May 1997 (1997-05-27) the whole document -----	9, 12, 15

38

INTERNATIONAL SEARCH REPORT
Information on patent family members

International Application No
PCT/US2004/037222

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 6559222	B1	06-05-2003	CA 2306478 A1 22-04-1999 EP 1023360 A2 02-08-2000 WO 9919369 A2 22-04-1999
X US 4745138	A	17-05-1988	NONE
US 3425977	A	04-02-1969	NONE
US 3862067	A	21-01-1975	BE 798169 A1 15-10-1973 DE 2318555 A1 18-10-1973 FR 2180073 A1 23-11-1973 GB 1413001 A 05-11-1975 IT 983008 B 31-10-1974 JP 49018124 A 18-02-1974 NL 7304875 A 16-10-1973 SE 380285 B 03-11-1975
US 5633307	A	27-05-1997	AT 178918 T 15-04-1999 AU 3640495 A 03-07-1996 CA 2204178 A1 20-06-1996 DE 69509119 D1 20-05-1999 DE 69509119 T2 23-12-1999 EP 0797607 A1 01-10-1997 ES 2133808 T3 16-09-1999 JP 10502700 T 10-03-1998 WO 9618668 A1 20-06-1996 US 6090881 A 18-07-2000

PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

To:

see form PCT/ISA/220

PCT

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY (PCT Rule 43bis.1)

Date of mailing
(day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference
see form PCT/ISA/220

FOR FURTHER ACTION
See paragraph 2 below

International application No.
PCT/US2004/037222

International filing date (day/month/year)
05.11.2004

Priority date (day/month/year)
06.11.2003

International Patent Classification (IPC) or both national classification and IPC
C08F8/00, C08F8/44

Applicant
SUN CHEMICAL CORPORATION

1. This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☒ Box No. VII Certain defects in the international application
- ☒ Box No. VIII Certain observations on the international application

2. FURTHER ACTION

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA"). However, this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1bis(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of three months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

3. For further details, see notes to Form PCT/ISA/220.

Name and mailing address of the ISA:



European Patent Office - P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk - Pays Bas
Tel. +31 70 340 - 2040 Tx: 31 651 epo nl
Fax: +31 70 340 - 3016

Authorized Officer

Droghetti, A

Telephone No. +31 70 340-4143



**WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING AUTHORITY**

International application No. *61*
PCT/US2004/037222

Box No. I Basis of the opinion

1. With regard to the **language**, this opinion has been established on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
☐ This opinion has been established on the basis of a translation from the original language into the following language , which is the language of a translation furnished for the purposes of international search (under Rules 12.3 and 23.1(b)).
2. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application and necessary to the claimed invention, this opinion has been established on the basis of:
 - a. type of material:
☐ a sequence listing
☐ table(s) related to the sequence listing
 - b. format of material:
☐ in written format
☐ in computer readable form
 - c. time of filing/furnishing:
☐ contained in the international application as filed.
☐ filed together with the international application in computer readable form.
☐ furnished subsequently to this Authority for the purposes of search.
3. ☐ In addition, in the case that more than one version or copy of a sequence listing and/or table relating thereto has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.
4. Additional comments:

62

Box No. V Reasoned statement under Rule 43b/s.1(a)(I) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	
	No: Claims	1-16
Inventive step (IS)	Yes: Claims	
	No: Claims	1-16
Industrial applicability (IA)	Yes: Claims	1-16
	No: Claims	

2. Citations and explanations

see separate sheet

Box No. VII Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Re Item V

Reference is made to the following document/s:

D1: US B 6559222 (cited by the applicant)
D2: US A 4745138
D3: US A 3425977
D4: US A 3862067
D5: US A 5633307

1. Novelty (Art. 33(2) PCT)

The subject-matter of claims 1-16 is not novel for the following reasons.

1.1. D1 discloses the same polymer composition for a coating composition comprising an ethylenically unsaturated acid resin, a neutralizing agent (e.g. ammonia) and water (see search report). Note that the only part in the claims, which appears as being novel with regard to D1, is the use of an ethyl. unsaturated amine as neutralizing agent.

Thus the subject-matter of claims 1-8, 10, 11, 13, 14, 16, is not novel in view of D1.

1.2. D2-D4 disclose curable coating composition comprising acid resins, a neutralizing agent (e.g. amines or ammonia) and water as claimed.

Thus the subject-matter of claims 1, 4-8, 11, 14 is not novel in view of D2-D4

1.3. In D5 a polymer composition comprising a polyamine with acrylic functional groups, an acid and water as claimed is disclosed.

A coating composition is prepared thereof.

Note that in the present application no specific method for preparing the composition is claimed. In claims 11-14, no defined technical features characterising the process are provided.

Thus the subject-matter of claims 9, 12, 15 is not novel in view of D2.

2. Inventive Step (Art. 33(3) PCT)

Being not novel the subject-matter of claims 1-16 cannot be regarded as inventive either.

3. The subject-matter of claims 1-16 meets the requirements of **Article 33(4) PCT**, with regard to **industrial applicability**.

Re Item VII

Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents D1, D3-D5 is not mentioned in the description, nor are these documents identified therein.

Re Item VIII (Art. 6 PCT)

1. In claim 1 the process features regarding the curing method (see "upon curing with an actinic radiation source") do not limit the process claim rendering the same unclear.

Furthermore the wording "offering the composition an increased crosslinked density" attempts to define the invention by the result-to-be-achieved.

In cl. 1 the expression "stereo-crosslinked" has no well-known or recognized meaning in the art.

This term is vague and unclear and leaves the reader in doubt as to the meaning of the technical features to which it refers, thereby rendering the definition of the subject-matter of said claim unclear, Article 6 PCT (the same applies to the expression "energy curable" in claims 11-16).

2. Although claims 1 and 10 have been drafted as separate independent claims, they appear to relate effectively to the same subject-matter and to differ from each other only with regard to the definition of the subject-matter for which protection is sought and in respect of the terminology used for the features of that subject-matter. The aforementioned claims therefore lack conciseness and as such do not meet the requirements of Article 6 PCT.

3. Claim 9,12,15 are not supported by the description as required by Article 6 PCT, as their scope is broader than justified by the description and drawings (see page 3). No specific hint to the basic resin as claimed can be found in the description and no process or article therefrom is mentioned either.

4. Claims 1,6,10-16 are not supported by the description.

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference (if desired) (12 characters maximum) C-574

Box No. I TITLE OF INVENTION WATER SOLUBLE ENERGY CURABLE STEREO-CROSSLINKABLE IONOMER COMPOSITIONS	
Box No. II APPLICANT ☐ This person is also inventor	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.) SUN CHEMICAL CORPORATION 222 Bridge Plaza South Fort Lee, New Jersey 07024 United States of America	Telephone No. (201) 224-4600 Facsimile No. (201) 224-2439 Teleprinter No. Applicant's registration No. with the
State (that is, country) of nationality: US	State (that is, country) of residence: US
This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of ☐ the United States of America only ☐ the States indicated in the Supplemental Box	
Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.) LINZER, Linzer 417 Seventh Street Carlstadt, NJ 07072	This person is: ☐ applicant only ☒ applicant and inventor ☐ inventor only (If this check-box is marked, do not fill in below.) Applicant's registration No. with the Office
State (that is, country) of nationality: US	State (that is, country) of residence: US
This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of ☒ the United States of America only ☐ the States indicated in the Supplemental Box	
☒ Further applicants and/or (further) inventors are indicated on a continuation sheet.	
Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE	
The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: ☒ agent ☐ common representative	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) PERSLEY, Sidney 222 Bridge Plaza South Fort Lee, New Jersey 07024 United States of America	Telephone No. (201) 224-4600 Facsimile No. (201) 224-2439 Teleprinter No. Agent's registration No. with the Office
☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.	

Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S) <i>If none of the following sub-boxes is used, this sheet should not be included in the request.</i>	
Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i> JONES, Richard M. 161 Greendale Drive Oak Ridge, New Jersey 07438 United States of America	This person is: ☐ applicant only ☒ applicant and inventor ☐ inventor only <i>(If this check-box is marked, do not fill in below.)</i> Applicant's registration No. with the
State <i>(that is, country)</i> of nationality: US	State <i>(that is, country)</i> of residence: US
This person is applicant ☐ all designated States ☐ all designated States except the United States of ☒ the United States of America only ☐ the States indicated in the Supplemental	
Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i>	This person is: ☐ applicant only ☐ applicant and inventor ☐ inventor only <i>(If this check-box is marked, do not fill in below.)</i> Applicant's registration No. with the Office
State <i>(that is, country)</i> of nationality:	State <i>(that is, country)</i> of residence:
This person is applicant for the ☐ all designated States ☐ all designated States except the United States of ☐ the United States of America only ☐ the States indicated in the Supplemental	
Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i>	This person is: ☐ applicant only ☐ applicant and inventor ☐ inventor only <i>(If this check-box is marked, do not fill in below.)</i> Applicant's registration No. with the
State <i>(that is, country)</i> of nationality:	State <i>(that is, country)</i> of residence:
This person is applicant for the ☐ all designated States ☐ all designated States except the United States of ☐ the United States of America only ☐ the States indicated in the Supplemental	
Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i>	This person is: ☐ applicant only ☐ applicant and inventor ☐ inventor only <i>(If this check-box is marked, do not fill in below.)</i> Applicant's registration No. with the
State <i>(that is, country)</i> of nationality:	State <i>(that is, country)</i> of residence:
This person is applicant for the ☐ all designated States ☐ all designated States except the United States of ☐ the United States of America only ☐ the States indicated in the Supplemental	
☐ Further applicants and/or (further) inventors are indicated on another continuation sheet.	

Box No. V DESIGNATIONS

The filing of this request constitutes under Rule 4.9(a), the designation of all Contracting States bound by the PCT on the international filing date, for the grant of every kind of protection available and, where applicable, for the grant of both regional and national patents.

However,

- ☐ DE Germany is not designated for any kind of national protection
- ☐ KR Republic of Korea is not designated for any kind of national protection
- ☐ RU Russian Federation is not designated for any kind of national protection

(The check-boxes above may be used to exclude (irrevocably) the designations concerned in order to avoid the ceasing of the effect, under the national law, of an earlier national application from which priority is claimed. See the Notes to Box No. V as to the consequences of such national law provisions in these and certain other States.)

Box No. VI PRIORITY CLAIM

The priority of the following earlier application(s) is hereby claimed:

Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country or Member	regional application: * regional Office	international application: receiving Office
item (1) 06/11/03	10/702,313	US		
item (2)				
item (3)				

- ☐ Further priority claims are indicated in the Supplemental Box.

The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of this international application is the receiving Office) identified above as:

- ☐ all items ☒ item (1) ☐ item (2) ☐ item (3) ☐ other, see Supplemental Box

* Where the earlier application is an ARIPO application, indicate at least one country party to the Paris Convention for the Protection of Industrial Property or one Member of the World Trade Organization for which that earlier application was filed (Rule 4.10(b)(ii)):

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):

ISA/ EPO

Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority):

Date (day/month/year) Number Country (or regional Office)

Box No. VIII DECLARATIONS

The following declarations are contained in Boxes Nos. VIII (i) to (v) (mark the applicable check-boxes below and indicate in the right column the number of each type of declaration):

		Number of declarations
☐ Box No. VIII (i)	Declaration as to the identity of the inventor	:
☐ Box No. VIII (ii)	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	:
☐ Box No. VIII (iii)	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	:
☐ Box No. VIII (iv)	Declaration of inventorship (only for the purposes of the designation of the United States of America)	:
☐ Box No. VIII (v)	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty:	:

Box No. IX CHECK LIST; LANGUAGE OF FILING																																																																						
This international application contains: (a) in paper form, the following number of sheets: request (including declaration sheets) : 4 description (excluding sequence listing and/or tables related thereto) : 13 claims : 3 abstract : 1 drawings : _____ Sub-total number of sheets 21 sequence listing : _____ tables related thereto : _____ <i>(for both, actual number of sheets if filed in paper form, whether or not also filed in computer readable form; see (c))</i> Total number of sheets : 21 (b) ☐ only in computer readable form (Section 801(a)(i)) (i) ☐ sequence listing (ii) ☐ tables related thereto (c) ☐ also in computer readable form (Section 801(a)(ii)) (i) ☐ sequence listing (ii) ☐ tables related thereto Type and number of carriers (diskette, CD-ROM, CD-R or other) on which are contained the ☐ sequence listing: _____ ☐ tables related thereto: _____ <i>(additional copies to be indicated under items 9(ii) and/or 10(ii), in right column)</i>	This international application is accompanied by the following item(s) (mark the applicable check-boxes below and indicate in right column the number of each item): <table style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 5%;">1.</td> <td style="width: 85%;">☒ fee calculation sheet</td> <td style="width: 10%; text-align: right;">:</td> <td style="width: 10%;"></td> </tr> <tr> <td>2.</td> <td>☐ original separate power of attorney</td> <td>:</td> <td></td> </tr> <tr> <td>3.</td> <td>☐ original general power of attorney</td> <td>:</td> <td></td> </tr> <tr> <td>4.</td> <td>☐ copy of general power of attorney; reference number, if any: _____</td> <td>:</td> <td></td> </tr> <tr> <td>5.</td> <td>☐ statement explaining lack of signature</td> <td>:</td> <td></td> </tr> <tr> <td>6.</td> <td>☐ priority document(s) identified in Box No. VI as item(s): _____</td> <td>:</td> <td></td> </tr> <tr> <td>7.</td> <td>☐ translation of international application into (language): _____</td> <td>:</td> <td></td> </tr> <tr> <td>8.</td> <td>☐ separate indications concerning deposited microorganism or other biological material</td> <td>:</td> <td></td> </tr> <tr> <td>9.</td> <td>☐ sequence listing in computer readable form (indicate type and number of carriers)</td> <td>:</td> <td></td> </tr> <tr> <td></td> <td>(i) ☐ copy submitted for the purposes of international search under Rule 13ter only (and not as part of the international application)</td> <td>:</td> <td></td> </tr> <tr> <td></td> <td>(ii) ☐ (only where check-box (b)(i) or (c)(i) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Rule 13ter</td> <td>:</td> <td></td> </tr> <tr> <td></td> <td>(iii) ☐ together with relevant statement as to the identity of the copy or copies with the sequence listing mentioned in left column</td> <td>:</td> <td></td> </tr> <tr> <td>10.</td> <td>☐ tables in computer readable form related to sequence listing (indicate type and number of carriers)</td> <td>:</td> <td></td> </tr> <tr> <td></td> <td>(i) ☐ copy submitted for the purposes of international search under Section 802(b-quater) only (and not as part of the international application)</td> <td>:</td> <td></td> </tr> <tr> <td></td> <td>(ii) ☐ (only where check-box (b)(ii) or (c)(ii) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Section 802(b-quater)</td> <td>:</td> <td></td> </tr> <tr> <td></td> <td>(iii) ☐ together with relevant statement as to the identity of the copy or copies with the tables mentioned in left column</td> <td>:</td> <td></td> </tr> <tr> <td>11.</td> <td>☐ other (specify): _____</td> <td>:</td> <td></td> </tr> </table>	1.	☒ fee calculation sheet	:		2.	☐ original separate power of attorney	:		3.	☐ original general power of attorney	:		4.	☐ copy of general power of attorney; reference number, if any: _____	:		5.	☐ statement explaining lack of signature	:		6.	☐ priority document(s) identified in Box No. VI as item(s): _____	:		7.	☐ translation of international application into (language): _____	:		8.	☐ separate indications concerning deposited microorganism or other biological material	:		9.	☐ sequence listing in computer readable form (indicate type and number of carriers)	:			(i) ☐ copy submitted for the purposes of international search under Rule 13ter only (and not as part of the international application)	:			(ii) ☐ (only where check-box (b)(i) or (c)(i) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Rule 13ter	:			(iii) ☐ together with relevant statement as to the identity of the copy or copies with the sequence listing mentioned in left column	:		10.	☐ tables in computer readable form related to sequence listing (indicate type and number of carriers)	:			(i) ☐ copy submitted for the purposes of international search under Section 802(b-quater) only (and not as part of the international application)	:			(ii) ☐ (only where check-box (b)(ii) or (c)(ii) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Section 802(b-quater)	:			(iii) ☐ together with relevant statement as to the identity of the copy or copies with the tables mentioned in left column	:		11.	☐ other (specify): _____	:		Number of items
1.	☒ fee calculation sheet	:																																																																				
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11.	☐ other (specify): _____	:																																																																				
Figure of the drawings which should accompany the abstract: _____	Language of filing of the international application: English																																																																					
Box No. X SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE <i>Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).</i> _____ Volker Linzer _____ Richard M. Jones																																																																						

For receiving Office use only	
1. Date of actual receipt of the purported international application: _____	2. Drawings: ☐ received: ☐ not received:
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application: _____	
4. Date of timely receipt of the required corrections under PCT Article 11(2): _____	
5. International Searching Authority (if two or more are competent): ISA/ _____	6. ☐ Transmittal of search copy delayed until search fee is paid
For International Bureau use only Date of receipt of the record copy by the International Bureau: _____	

PCT

FEE CALCULATION SHEET

Annex to the Request

For receiving Office use only

International Application No.

Date stamp of the receiving Office

Applicant's or agent's
file reference

C-574

Applicant

SUN CHEMICAL CORPORATION

CALCULATION OF PRESCRIBED FEES

1. TRANSMITTAL FEE 300.00 T

2. SEARCH FEE 1,920.00 S

International search to be carried out by EPO
(If two or more International Searching Authorities are competent to carry out the international search, indicate the name of the Authority which is chosen to carry out the international search.)

3. INTERNATIONAL FILING FEE

Where item (b) and/or (c) of Box No. IX apply, enter Sub-total number of sheets } 19
Where item (b) and (c) of Box No. IX do not apply, enter Total number of sheets }

i1 first 30 sheets 1,053.00 i1

i2 0 x = 0.00 i2
number of sheets in excess of 30 fee per sheet

i3 additional component (only if sequence listing and/or tables related thereto are filed in computer readable form under Section 801(a)(i), or both in that form and on paper, under Section 801(a)(ii):

400 x = i3
fee per sheet

Add amounts entered at i1, i2 and i3 and enter total at I 1,053.00 I

(Applicants from certain States are entitled to a reduction of 75% of the international filing fee. Where the applicant is (or all applicants are) so entitled,

4. FEE FOR PRIORITY DOCUMENT (if applicable) 20.00 P

5. TOTAL FEES PAYABLE 3,293.00

Add amounts entered at T, S, I and P, and enter total in the TOTAL box

TOTAL

MODE OF PAYMENT

- ☒ authorization to charge deposit account (see below) ☐ postal money order ☐ cash ☐ coupons
☐ cheque ☐ bank draft ☐ revenue stamps ☐ other (specify):

AUTHORIZATION TO CHARGE (OR CREDIT) DEPOSIT ACCOUNT

(This mode of payment may not be available at all receiving Offices)

- ☒ Authorization to charge the total fees indicated above.
☒ (This check-box may be marked only if the conditions for deposit accounts of the receiving Office so permit) Authorization to charge any deficiency or credit any overpayment in the total fees indicated above.
☒ Authorization to charge the fee for priority document.

Receiving Office: US

Deposit Account No.: 19-4968

Date: November 5, 2004

Name: Sidney Persley

Signature: Sidney Persley

PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU



PCT

NOTIFICATION OF RECEIPT OF
RECORD COPY

(PCT Rule 24.2(a))

Handwritten signature and date: JAN 18 2005

To:

PERSLEY, Sidney
222 Bridge Plaza South
Fort Lee, NJ 07024
United States of America

Handwritten '30'

Date of mailing (day/month/year) 14 January 2005 (14.01.2005)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference C-574	International application No. PCT/US2004/037222

The applicant is hereby notified that the International Bureau has received the record copy of the international application as detailed below.

Name(s) of the applicant(s) and State(s) for which they are applicants:

SUN CHEMICAL CORPORATION (for all designated States except US)
LINZER, Linzer et al (for US)

International filing date : 05 November 2004 (05.11.2004)
Priority date(s) claimed : 06 November 2003 (06.11.2003)
Date of receipt of the record copy
by the International Bureau : 03 January 2005 (03.01.2005)
List of designated Offices :

AP : BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW
EA : AM, AZ, BA, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM,
EP : AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LU, MC, NL, PL, PT, RO, SE, SI,
SK, TR
OA : BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
National : AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM,
DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS,
LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK,
SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No. (41-22) 338.71.30	Authorized officer: R. Meach (Fax 338-71-30) Telephone No. (41-22) 338 9266
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Continuation of Form PCT/IB/301

NOTIFICATION OF RECEIPT OF RECORD COPY

71

Date of mailing (day/month/year) 14 January 2005 (14.01.2005)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference C-574	International application No. PCT/US2004/037222

ATTENTION

The applicant should carefully check the data appearing in this Notification. In case of any discrepancy between these data and the indications in the international application, the applicant should immediately inform the International Bureau.

In addition, the applicant's attention is drawn to the information contained in the Annex, relating to:

- ☒ time limits for entry into the national phase - see updated important information (as of April 2002)
- ☒ requirements regarding priority documents (if applicable)

A copy of this Notification is being sent to the receiving Office and to the International Searching Authority.

INFORMATION ON TIME LIMITS FOR ENTERING THE NATIONAL PHASE

The applicant is reminded that the "national phase" must be entered before each of the designated Offices indicated on the cover sheet of this Notification by paying national fees and furnishing translations, as prescribed by Articles 22 and 39 and the applicable national laws. In addition, the applicant may also have to comply with other special requirements applicable in certain Offices. It is the applicant's responsibility to ensure the necessary steps to enter the national phase are taken in a timely fashion. Most Offices do not issue reminders to applicants in connection with the entry into the national phase.

The applicable time limit for entering the national phase will, subject to what is said in the following paragraph, be **30 MONTHS** from the priority date, not only in respect of any elected Office if a demand for international preliminary examination is filed before the expiration of 19 months from the priority date (see Article 39(1)), but also in respect of any designated Office, in the absence of filing of such demand, where Article 22(1) as modified with effect from 1 April 2002 applies in respect of that designated Office. For further details, see PCT Gazette No. 44/2001 of 1 November 2001, pages 19926, 19932 and 19934, as well as the PCT Newsletter, October and November 2001 and February 2002 issues.

In practice, time limits other than the 30-month time limit will continue to apply, for various periods of time, in respect of certain designated or elected Offices. For regular updates on the applicable time limits (20, 21, 30 or 31 months, or other time limit), Office by Office, refer to the PCT Gazette ("Section IV" part published on a weekly basis), to the PCT Newsletter (on a monthly basis) and to the relevant National Chapters in Volume II of the PCT Applicant's Guide (the paper version of which is updated usually twice a year and the Internet version of which is updated usually on a weekly basis). Finally, a cumulative table of all applicable time limits for entering the national phase is available from WIPO's Internet site, via links from various pages the site including those of the Gazette, Newsletter and Guide, at <http://www.wipo.int/pct/en/index.html>.

Information about the requirements for filing a demand for international preliminary examination is set out in the PCT Applicant's Guide, Volume I/A, Chapter IX. Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination (at present, all PCT Contracting States are bound by Chapter II).

REQUIREMENTS REGARDING PRIORITY DOCUMENTS

For applicants who have not yet complied with the requirements regarding priority documents, the following is recalled.

Where the priority of an earlier national, regional or international application is claimed, the applicant must submit a copy of the said earlier application, certified by the authority with which it was filed ("the priority document") to the receiving Office (which will transmit it to the International Bureau) or directly to the International Bureau, before the expiration of 16 months from the priority date, provided that any such priority document may still be submitted to the International Bureau before that date of international publication of the international application, in which case that document will be considered to have been received by the International Bureau on the last day of the 16-month time limit (Rule 17.1(a)).

Where the priority document is issued by the receiving Office, the applicant may, instead of submitting the priority document, request the receiving Office to prepare and transmit the priority document to the International Bureau. Such request must be made before the expiration of the 16-month time limit and may be subjected by the receiving Office to the payment of a fee (Rule 17.1(b)).

If the priority document concerned is not submitted to the International Bureau or if the request to the receiving Office to prepare and transmit the priority document has not been made (and the corresponding fee, if any, paid) within the applicable time limit indicated under the preceding paragraphs, any designated State may disregard the priority claim, provided that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within the time limit which is reasonable under the circumstances (Rule 17.1(c)).

Where several priorities are claimed, the priority date to be considered for the purposes of computing the 16-month time limit (and all other PCT time limits) is the filing date of the earliest application whose priority is claimed (Article 2(xi)(b)).

From the INTERNATIONAL BUREAU

PCTNOTIFICATION CONCERNING
SUBMISSION OR TRANSMITTAL
OF PRIORITY DOCUMENT

(PCT Administrative Instructions, Section 411)

To:

PERSLEY, Sidney
222 Bridge Plaza South
Fort Lee, NJ 07024
ETATS-UNIS D'AMERIQUE

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Applicant SUN CHEMICAL CORPORATION et al	

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<u>Priority date</u>	<u>Priority application No.</u>	<u>Country or regional Office or PCT receiving Office</u>	<u>Date of receipt of priority document</u>
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The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer Olaiz Alicia
Facsimile No. +41 22 740 14 35	Facsimile No. +41 22 338 71 30 Telephone No. +41 22 338 9288

From the INTERNATIONAL BUREAU

PCT

SECOND AND SUPPLEMENTARY NOTICE
INFORMING THE APPLICANT OF THE
COMMUNICATION OF THE INTERNATIONAL
APPLICATION (TO DESIGNATED OFFICES
WHICH APPLY THE 30 MONTH TIME
LIMIT UNDER ARTICLE 22(1))

(PCT Rule 47.1(c))

To:

PERSLEY, Sidney
c/o Sun Chemical Corporation
35 Waterview Blvd.
Parsippany, NJ 07054
ETATS-UNIS D'AMERIQUE

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79

Date of mailing (day/month/year) 09 March 2006 (09.03.2006)		
Applicant's or agent's file reference C-574		IMPORTANT NOTICE
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Applicant SUN CHEMICAL CORPORATION et al		

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26 May 2005 (26.05.2005)

AU, AZ, BY, CN, CO, DZ, EP, HU, KG, KP, KR, MD, MK, MZ, NA, PG, RU, SY, TM, US

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34, chemin des Colombettes
1211 Geneva 20, Switzerland

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Simin Baharlou

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(71) Applicant (for all designated States except US): **SUN CHEMICAL CORPORATION** [US/US]; 222 Bridge Plaza South, Fort Lee, NJ 07024 (US).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **LINZER, Linzer** [US/US]; 417 Seventh Street, Carlstadt, NJ 07072 (US). **JONES, Richard, M.** [US/US]; 161 Greendale Drive, Oak Ridge, NJ 07438 (US).

(74) Agent: **PERSLEY, Sidney**; 222 Bridge Plaza South, Fort Lee, NJ 07024 (US).

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(54) Title: WATER SOLUBLE ENERGY CURABLE STEREO-CROSSLINKABLE IONOMER COMPOSITIONS

(57) Abstract: A homogenous, aqueous, energy curable, stereo cross linkable ionomer containing coating composition and a method of preparing same.

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WATER SOLUBLE ENERGY CURABLE STEREO-CROSSLINKABLE IONOMER COMPOSITIONS

76

FIELD OF THE INVENTION

This invention relates to the synthesis and use of water soluble energy curable stereo crosslinkable ionomers used in the manufacture of coated and printed materials.

BACKGROUND OF THE INVENTION

Ionomers are polymeric compounds carrying electronic charges within the polymer chain. Ionomers build outstanding properties like hardness and solvent resistance when two or more such polymeric chains create a ladder-type structure by salt formation. The ionic complexation of identical (e.g., bridged by a higher-valent counterion) or opposing charges (e.g., acid/base neutralization of amine-functional with carboxyl-functional polymeric species) on the polymer chains results in additional crosslinks, the number per unit volume of which determines the mechanical strength of the resulting solid. The more commonly used ionic complexation route typically proceeds through use of a more stable complex of the bridging counterion with a volatile material (e.g. ammonia) to allow blending in the liquid state followed by later crosslinking upon drying to a solid. The acid/base neutralization of two oppositely charged ionomers to create crosslinking, in contrast, is largely impractical due to the impossibility of blending these materials to generate anything other than an intractable crosslinked solid.

Description of Related Art

The photopolymerization approach to ionomer crosslinking is not novel. U.S. Patents 6,281,271 and 6,017,982 disclose the energy curing of ethylenically unsaturated ions to build covalent molecular weight in-situ and to trigger crosslinking via ionic complexation of identical charges on two or more ionomer chains, in the presence of water and a divalent metaloxide. U.S. Patent 6,180,040 teaches the formation of an ionomer via photopolymerization using energy curable compositions of ionic monomers such as metal acrylic carboxylates copolymerized with polybutadiene resins. These compounds form ionomers upon polymerization and are bridged over a metal complex in a second (vulcanization) step. In neither of these examples is the bridging ion an organic polymer, either preexisting or formed by in-situ polymerization. Fundamentally,

the ionic complexation routes illustrated in the above references yield materials with regular, repeating crosslink patterns and compact ionic structures. X2

Ionomers that crosslink only over ethylenically unsaturated moieties (i.e., do not employ either bridging ions or acid/base combinations of separate, oppositely charged ionomers or monomers) are commonly used in coatings and photoresists. These compounds are usually water soluble polymers carrying ethylenically unsaturated groups in or grafted onto the polymer chain of the single-chain ionomer. Examples of these polymer types are neutralized acrylics described in U.S. Patent 4,275,142; styrene-maleic anhydride described in U.S. Patent 3,825,430 and 4,401,793; and polyester, or urethane polymer salts described in U.S. Patents 6,207,346 and 5,554,712. Since no use is made of the ionic groups to create additional crosslinks, cured films made from these ionomers are often structurally weakened by the existence of ionic charges, as these sites retain moisture, which plasticizes the cured final polymer (e.g., they show lower rub resistance to water compared to uncharged polymers).

U.S. Patents 4,745,138; 5,868,605 and 6,099,415 disclose the use of chemically similar but non-neutralized resins in energy curing compositions. However, these patents do not teach the formation of blends with resins, oligomers, or monomers that could potentially neutralize an energy curable resin in a way that might crosslink the polymer. In addition, the viscosity of these non-neutralized resins is typically high. To bring them to application viscosity they must be diluted with either a large amount of a low viscosity reactive monomer or with a solvent.

SUMMARY OF THE INVENTION

The invention is the formation and use of water soluble energy curable ionomers that form a stereo crosslinked network upon curing over ethylenically unsaturated and polyionic sites. As a result, energy curable stereo-crosslinkable ionomers are produced that deliver a low viscosity liquid and create a cured solid film having superior mechanical and solvent resistance properties along with good adhesion to difficult substrates. The materials also resist cracking and flaking, and offer improve gloss and rub resistance, and enhanced coverage when compared to existing materials used in formulating paints, inks, and coatings.

78

The present invention shows how in-situ photopolymerization can be used to generate the opposing charge type of polymer from low-viscosity liquid monomer/oligomer blends that have utility in the manufacture of coated and printed materials. The structures formed in the present invention are random, largely amorphous, three-dimensional networks of opposing charge polymers with control over the rigidity of the crosslink. The invention employs lower molecular weight oligomeric resins and water as solvent to reduce viscosity and accelerate cure at zero volatile organic content (VOC). Cure occurs in the presence of the water, and the dissolved water is allowed to concurrently dry without application of additional energy to give cured structures that are surprisingly not sensitive to water. The oligomeric resins are neutralized with ethylenically unsaturated polyamines to form water-soluble resin salts. In some instances these salts are liquids (low melting solids), but more generally they require a water content above 10 % to be fluid. In most instances, in order to provide useful viscosity, the water content will be above 30%.

In water based energy curable compositions, viscosity reducing monomeric compounds, typically employed in energy curable compositions are replaced with water. There are two fundamentally different technologies used in this field. One derives from ethylenically unsaturated water based emulsions, which are dried before curing. The other is based on partially soluble energy curable material where the curing reaction is carried out in solution and does not necessarily include a drying step before cure. The precursors employed in the present invention are water soluble at least partially water-soluble, a state that is obtained from the use of truly water soluble monomers and oligomers in admixture with the required ionic materials.

Ethylenically Unsaturated Resin

The water soluble ethyleneically unsaturated oligomeric or polymeric resin may have acid functional groups (e.g. carboxylic acid groups) which are partially or totally neutralized with a base (e.g., an amine) to form a water soluble resin salt. Alternatively, the resin may have basic functional groups (e.g. amino groups) which are partially or totally neutralized with an acid (e.g. a carboxylic acid) to form a water soluble resin salt. A preferred embodiment is a neutralization product, where a water soluble, acrylated, resin salt is formed from an ethylenically unsaturated energy curable resin containing acrylic groups, methacrylic groups or a combination thereof; and carboxylic acid

functional groups, neutralized by a base. While a more preferred resin salt product is a neutralization product prepared from an ethylenically unsaturated amine and a polyanionic resin. Suitable examples of a polyanionic resin are polyacrylic or styrene-maleic anhydride copolymers, containing carboxyl groups, and having an acid value of at least 80 (mg KOH per 100 g polymer). Commercially available examples of such resin are Carboset GA-1167 from BF Goodrich; Joncryl 690 from SC Johnson; and SMA 1000 from ELF Atochem. It is preferred that the polyanionic resin be partially esterified via a polymer analog reaction to tailor the final properties of the compound and the final product of compositions containing the compound (propanol, isopropanol, stearic alcohol, polypropylene glycol) but that such resin have the same acid number of 80. A preferred modification uses an ethylenically unsaturated alcohol to form an ethylenically unsaturated polyanionic resin containing at least two such functions per molecule. The resin is then neutralized to a pH of at least 5.5 with an ethylenically unsaturated amine or a mixture such amine and ammonia or other caustic component. If there is no ethylenically unsaturated content in the polyanionic resin, then the tertiary amine should be ethylenically unsaturated. If the polyanionic resin is ethylenically unsaturated, then the tertiary amine may be saturated provided that it contains at least two amine groups per molecule. However, it is most preferred that both components of the resin be ethylenically unsaturated.

A particularly preferred energy curable resin is a styrene/maleic anhydride copolymer partially esterified with a hydroxy alkyl acrylate or methacrylate. The hydroxy alkyl acrylate or methacrylate preferably of such resin is preferably hydroxy butyl acrylate or methacrylate. A partially esterified styrene/maleic anhydride copolymer may be neutralized without further modification or it may be further partially esterified with an alkanol such as butanol, propanol, ethanol and the like.

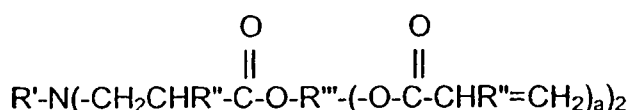
The acidic or carboxylic acid (anhydride) groups of the energy curable resin are partially or totally neutralized to provide a resin having the desired range of water solubility while retaining complete miscibility with other water soluble resins.

The resins used to form the compositions of the present invention while having an acid number of at least 80 have a molecular weight between 1,000 and 25,000; and more preferably between 1,000 and 10,000; and most preferably between 1,000 and 5,000.

80

Any basic compound (e.g., alkali metal hydroxides such as calcium hydroxide, potassium, hydroxide and sodium hydroxide, ammonia, amines, etc.) may be used to neutralize the acidic groups of the resin. Preferred are ammonia, amines or combinations thereof. Even more preferred is amines, while more particularly preferred is an ethylenically unsaturated tertiary amine. By employing ethylenically unsaturated tertiary amines as the neutralizing agent, the acidic groups on the energy curable resin are totally neutralized to form a water soluble resin having additional polymerizable ethylenic groups. The use of ethylenically unsaturated tertiary amines as the neutralizing agent further allows the acid groups on the resin to be totally neutralized which aids in the formation of the stereo cross-linkable water soluble ionomers of the present invention.

The ethylenically unsaturated tertiary amine neutralizing agent of the present invention has the formula:



wherein R' is a short chain hydrocarbon group; R'' is H or a methyl group; and R''' is selected from the group consisting of C₁-C₂₀ alkyl, C₁-C₂₀ aralkyl, C₁-C₂₀ alkyl substituted aralkyl and C₁-C₂₀ oxyalkylated derivatives; and a is 1, 2, or 3.

Preferably the ethylenically unsaturated tertiary amine is a Michael Addition product of a primary or secondary amine (e.g., an alkyl amine) and two acrylic esters, wherein each of the acrylic esters contains two or more acrylate or methacrylate groups (e.g., wherein the acrylic ester is an acrylate ester or methacrylate ester of an alkane diol, a polyether diol, a glycol, a glycerol). The primary or secondary amine may be selected, for example, from ethyl amines, ethanol amines, diethanol amines and hexamethylene imines and combinations thereof. The acrylic ester may be selected from hexanediol diacrylate, dipropylene glycol diacrylate (DPGDA), tripropylene glycol diacrylate (TPGDA), trimethylolpropane triacrylate (TMPTA), pentaerythritol triacrylate (PETA) and combinations thereof. Examples of commercially available acrylic functional amines are Laromer 8996 (available from BASF); Ebecryl 7100 (available from UCB); Suncure 175 (available from Reichhold); and Ebecryl P-104 (available from UCB).

Stereo Cross-Linkable Ionomer

It should be noted that the monomeric/oligomeric nature of the ionomer components results in a random distribution of the cationic and anionic charges within the ionomer network (see Figure 1). This differs from existing preformed polymeric ionomer pairs in which one of the pair of polymers contains cationic charges while the other contains anionic charges (see Figure 2). In the present invention, stereo cross-linkable is defined as the ability of the oligomeric ionomers to randomly polymerize in-situ via two different mechanisms. The first mechanism being a covalent free radical photopolymerization of the resin. The second mechanism being an ionic cross-link between the acidic and basic functional groups of the resin in multiple dimensions at the same time, to form a highly crosslinked polymeric network of infinite molecular mass. For example, the ethylenically unsaturated tertiary amine neutralizing agent provides the counter ion for the acidic ethylenically unsaturated resin, which allows the ionomer formed, to stereo polymerize during photoreaction (via energy curing) and form an additional cross-linked network over the ethylenically unsaturated groups as well as over the ionic structure of the resin. Therefore, unlike other water based energy curable resin technology, where the resistance properties to be imparted by the cured resin composition depend on, and are a function of, the evaporation of the base (e.g. ammonia), for example, which shifts the acid base equilibrium in the post cure composition, here for example, the ethylenically unsaturated tertiary amine neutralizing base, and neutralized resin form an additional cross-linked network instantly on both sides of the ionomer.

Upon irradiation, either or both of the ions formed from the two oligomeric material formations increase in molecular weight by addition polymerization. The use of (meth)acrylic functional, carboxylic polymeric, and tertiary amine oligomeric resins in an energy curing mechanism results in the formation of a crosslinked ionomer with a ladder structure via the formation of covalent bonds as well as opposing ionic bonds. It is essential that one oligomeric material polymerizes but it is preferred that they both polymerize. After polymerization, a highly crosslinked structure is formed showing exceptional properties over conventional and "water-compatible" energy curable materials existing in the prior art. The use of the present composition in energy curable leads to improved cure, adhesion, hardness, mechanical and solvent resistance properties.

The water solubility of the polymeric resin salt of the present invention makes it especially suitable for water based energy curing processes while the organic acid/base

crosslinking works against the tendency of ionic polymers to become mechanically weaker upon absorption of water. The control of the molecular weight between acidic and basic sites on separate oligomers allows for the formation of highly crosslinked materials which show much less brittle failure than expected for crosslinked ionomers.

For the synthesis of the resin salt three processes can be used. The first process starts from a solid carboxylic resin, which will be diluted in tertiary acrylic amine and optionally, water. The second process starts with the polymer analog reaction of alcohols with the styrene maleic anhydride resin in a solvent. In a solvent stripping step, the resin will be neutralized and diluted with tertiary acrylic amine. In the third process the solvent of the previous described ester modified resin will be azeotropically distilled with water and partially neutralized with ammonia. Afterward the resin is further neutralized with acrylated amine at 60°C

Example 1 (Comparative)

SMA-1000 (40 grams, available from Atofina) solid is slurried into water (50 grams) and neutralized to pH 6.5 with concentrated NH_4OH (available from Fisher Scientific). The resulting solution was 43 % in solids content and had a 31 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

Example 2

SMA-1000 (40 grams, available from Atofina) solids is slurried into water (50 grams) and neutralized to pH 6.5 with di(2-hydroxyethyl)methyl amine (15 grams, available from Aldrich) with heating to 60 °C for 12 hours. The resulting solution was 50 % solids in content and had a 30 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

Example 3

SMA-1000 (35 grams, available from Atofina) solids is slurried into water (50 grams) and neutralized to pH 4.5 with concentrated NH_4OH (5 grams, available from Fisher Scientific) followed by addition of acrylated amine 16-101 (10 grams, available from Reichhold) and heating to 60 °C for 4 hours. After cooling, the resulting solution was corrected to pH 6.5 with additional concentrated ammonia and measured to be 48 % in solids content and had a 48 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

23

Example 4 (Comparative)

As described in US Patent 6,559,222, polymeric resin salt, styrene maleic anhydride copolymer (165 grams) having an acid number of 480 and an average molecular weight of 1000 were added together under agitation to methyl isobutyl ketone (MIBK, 120 grams) . The two materials were then heated to approximately 95-110 degrees C. over 1 to 2 hours under a nitrogen blanket. Next, N, N-dimethylbenzyl amine (0.8 grams) and a monofunctional alcohol (18 grams) such as n-propanol, ethanol or octadecanol were then added to form a polymeric mixture having an acid number between 200 to 210. The nitrogen blanket was then removed and 4-methoxyphenol (0.12 grams) and N, N-dimethylbenzylamine (0.36 grams) were added. Over a period of time, for example 60 to 90 minutes, a hydroxy-functional acrylate such as 4-hydroxybutyl acrylate (55.80 grams) or 2-hydroxy-ethyl acrylate was then added until the acid number of the polymeric mixture is between 130 to 140. The polymeric mixture was then distilled and 4-methoxyphenol (0.12 grams) was added along with ammonium hydroxide (27.90 grams) and deionized water (327.8 grams). The mixture was then heated, for example to 99 degrees C. The MIBK and water were then removed by distillation. When all of the MIBK had been removed, the water is returned to the mixture as a water/ammonia distillate. This material was prepared as a 37 % in solids content in water and neutralized to pH 6.5 with ammonia yielding 23 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

Example 5

To the resin salt solution (108 grams) as prepared in Example 4 at pH 4.5 (containing 37% resin solids in water prior to the final addition of neutralizing base described in US Patent 6,559,222) at 60 °C was added di(2-hydroxyethyl)methyl amine (10 grams, Aldrich). After cooling, the resulting solution had a pH of 6.5 and was 43 % in solids content and 20 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

Example 6

To resin salt solution (100 grams) as prepared in Example 4 at pH 4.5 (containing 37% resin solids in water prior to the final addition of neutralizing base described in US Patent 6,559,222) at 60 °C was added acrylated amine 16-101 (15 grams, Reichhold) over four hours. Upon cooling, the resulting solution was corrected to pH 6.5 with concentrated ammonia and measured to be 48 % in solids content and had a 37 Pa.s. viscosity at 10 s⁻¹ at 40 °C.

84

Example 7 (Comparative)

Laromer 8765 (20 grams, available from BASF Corporation, Mount Olive, NJ) was added to the resin salt solution (25 grams) as prepared in Example 1. Irgacure 2959 (1.5 grams, available from Ciba) was then added to this solution. To complete the solution for coating, water (3 grams) and TegoRad 2200N (0.5 grams available from TegoRad Corporation) were added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of coating solution was 0.35 Pa.s. at 25 °C.

Example 8

Laromer 8765 (17.5 grams, available from BASF) followed by Irgacure 2959 (1.5 grams, available from Ciba) were added with stirring to resin salt solution in water (27 grams) as prepared in Example 2. water (3.5 grams) and TegoRad 2200N (0.5 grams available from TegoRad Corporation) were then added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of coating solution was 0.38 Pa.s. at 25 °C.

Example 9

Laromer 8765 (17.5 grams, BASF) followed by Irgacure 2959 (1.5 grams, available from Ciba) were added with stirring to resin salt solution in water (30.5 grams) as prepared in Example 3. TegoRad 2200N (0.5 grams available from TegoRad Corporation) was then added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of coating solution was 0.52 Pa.s. at 25 °C.

Example 10 (Comparative)

Laromer 8765 (17.5 grams, BASF) followed by Irgacure 2959 (1.5 grams, available from Ciba) were added with stirring to resin salt solution in water (30.5 grams) as prepared in Example 4. TegoRad 2200N (0.5 grams, available from TegoRad Corporation) was then added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of the coating solution was 0.23 Pa.s. at 25 °C.

Example 11

Laromer 8765 (17.5 grams, available from BASF) followed by Irgacure 2959 (1.5 grams, available from Ciba) were added with stirring to resin salt solution in water (30.5 grams) as prepared in Example 5. TegoRad 2200N (0.5 grams, available from TegoRad Corporation) was then added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of the coating solution was 0.28 Pa.s. at 25 °C.

Example 12

Laromer 8765 (18.0 grams, available from BASF) followed by Irgacure 2959 (1.5 grams, available from Ciba) were added with stirring to resin salt solution in water (30 grams) as prepared in Example 6. TegoRad 2200N (0.5 grams, available from TegoRad Corporation) was then added with stirring, and the solution set aside for 12 hours to clear the entrained air before coating and curing. The viscosity of the coating solution was 0.25 Pa.s. at 25 °C.

Example 13

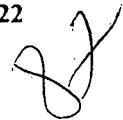
The coating solutions described in Examples 7 to 12 were applied by #3 and #5 wire-wound rods to Uncoated Leneta N2A charts (Leneta is a product and trademark of The Leneta Company, 15 Whitney Rd, Mahwah, NJ). Immediately following coating, the wet film was cured by passing under 650 mJ/cm² of UV light (two medium pressure Hg lamps at 300 W/in each, 200 fpm on an RPC Industries processor) in air. The resulting cured surfaces were conditioned at 75 °F and 48 % Relative Humidity for one day and the following measurements taken. Gloss was measured at 60-degree angle using a type DIN Geproft 4501 meter from BYK Gardner parallel to the coating direction. The rub resistance (an methyl ethyl ketone (MEK) rub and water rub test) was determined by wetting the cured coating surface and employing light finger pressure to rub the coating off as detected by the exposure of the underlying ink. The number of complete back-and-forth cycles required were recorded. Coating adhesion was measured by taking a convenient length of 610 tape (available from 3M Co., St. Paul, MN), laminating the tape to the cured surface under finger pressure, then lifting the tape from the surface in one rapid motion at right angle to the coated surface. The adhesion was rated a pass when the coating remained completely intact and adhered to the substrate following tape peel. The coating weight was determined gravimetrically by the difference in weight between a 10 cm X 10 cm piece cut from the coated area and an

identical size piece cut from a similar area of uncoated stock. Each measurement reported in Table 1 below is normalized to the same dry coating weight (4 g/m²). 

Table 1

<u>Example</u>	<u>Gloss</u>	<u>MEK Rub</u>	<u>Water Rub</u>	<u>Adhesion</u>
7 (comparative)	65	6	5	fail
10 (comparative)	89	12	18	pass
11	91	12	16	pass
12	95	35	20	pass

Those skilled in the art having the benefit of the teachings of the present invention as hereinabove set forth, can effect numerous modifications thereto. These modifications are to be construed as being encompassed within the scope of the present invention as set forth in the appended claims.

CLAIMS

What we claim is:

1. An aqueous, energy curable, homogenous, composition comprising the neutralization product of
 - (a) an ethylenically unsaturated acidic resin containing carboxylic acid, acrylic functional groups, methacrylic functional groups or a combination thereof, and
 - (b) an ammonia, amine, alkali metal hydroxide or a combination thereof; in
 - (c) water;where upon curing with an actinic radiation source, a stereo-crosslinked ionomer forms offering the composition an increased cross-linked density.
2. The composition of Claim 1 wherein the, ethylenically unsaturated resin is a styrene/maleic anhydride copolymer, partially esterified with a hydroxy alkyl acrylate or methacrylate functional group.
3. The composition of Claim 2 wherein the partially esterified styrene/maleic anhydride copolymer is further esterified with an alcohol group.
4. The composition of Claim 1 wherein the neutralizing agent is ammonia, ethylenically unsaturated amine, or a combination thereof.
5. The composition of Claim 4 wherein the neutralizing agent is ammonia, ethylenically unsaturated tertiary amine, or a combination thereof
6. The composition according to Claim 1, wherein the ethylenically unsaturated resin has an acid number of at least 80 and a weight average molecular weight between 1,000 and 50,000.
7. The composition according to Claim 6, wherein the ethylenically unsaturated resin has an acid number of at least 80 and a weight average molecular weight between 1,000 and 25,000.

8. The composition according to Claim 7, wherein the ethylenically unsaturated resin has an acid number of at least 80 and a weight average molecular weight between 1,000 and 10,000.
9. An aqueous, energy curable, homogenous, composition comprising:
the neutralization product of
(d) an ethylenically unsaturated basic resin containing amine, acrylic functional groups, methacrylic functional groups or a combination thereof and
(e) an acid; in
(f) water,
where upon curing with an actinic radiation source, a stereo-crosslinked ionomer forms offering the composition an increased cross-linked density.
10. An aqueous, energy curable, homogenous, composition comprising:
the neutralization product of
(a) an ethylenically unsaturated styrene/maleic anhydride copolymer acidic resin, partially esterified with a hydroxy alkyl acrylate, further esterified by an alcohol, containing carboxylic acidic functional groups; and
(b) an ammonia, ethylenically unsaturated tertiary amine, or a combination thereof; in
(c) water,
where upon curing with an actinic radiation source, a stereo-crosslinked ionomer forms offering the composition an increased cross-linked density.
11. A method for preparing an energy curable coating comprising employing the composition of Claim 1.
12. A method for preparing an energy curable coating comprising employing the composition of Claim 9.
13. A method for preparing an energy curable coating comprising employing the composition of Claim 10.
14. An energy curable coating comprising the composition of Claim 1.

15. An energy curable coating comprising the composition of Claim 9.

16. An energy curable coating comprising the composition of Claim 10.

29

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US2004/037222

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C08F8/00 C08F8/44

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 7 C08F C09D

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)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 559 222 B1 (ROONEY JOHN ET AL) 6 May 2003 (2003-05-06) cited in the application the whole document	1-8, 10, 11, 13, 14, 16
X	US 4 745 138 A (THANAWALLA ET AL) 17 May 1988 (1988-05-17) cited in the application the whole document	1, 4-8, 11, 14
X	US 3 425 977 A (MARTIN SKOULTCHI ET AL) 4 February 1969 (1969-02-04) the whole document	1, 4-8, 11, 14
X	US 3 862 067 A (MOTIER ET AL) 21 January 1975 (1975-01-21) the whole document	1, 4-8, 11, 14
	-/--	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *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
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- * & * document member of the same patent family

Date of the actual completion of the international search

10 March 2005

Date of mailing of the international search report

24/03/2005

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Droghetti, A

INTERNATIONAL SEARCH REPORT

Inter ☐ National Application No
PCT/US2004/037222

91

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 633 307 A (DAS ET AL) 27 May 1997 (1997-05-27) the whole document -----	9, 12, 15

INTERNATIONAL SEARCH REPORT

information on patent family members

Inter ☐ National Application No
PCT/US2004/037222

92

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 6559222	B1	06-05-2003	CA 2306478 A1 EP 1023360 A2 WO 9919369 A2	22-04-1999 02-08-2000 22-04-1999
US 4745138	A	17-05-1988	NONE	
US 3425977	A	04-02-1969	NONE	
US 3862067	A	21-01-1975	BE 798169 A1 DE 2318555 A1 FR 2180073 A1 GB 1413001 A IT 983008 B JP 49018124 A NL 7304875 A SE 380285 B	15-10-1973 18-10-1973 23-11-1973 05-11-1975 31-10-1974 18-02-1974 16-10-1973 03-11-1975
US 5633307	A	27-05-1997	AT 178918 T AU 3640495 A CA 2204178 A1 DE 69509119 D1 DE 69509119 T2 EP 0797607 A1 ES 2133808 T3 JP 10502700 T WO 9618668 A1 US 6090881 A	15-04-1999 03-07-1996 20-06-1996 20-05-1999 23-12-1999 01-10-1997 16-09-1999 10-03-1998 20-06-1996 18-07-2000

TRANSMITTAL LETTER TO THE UNITED STATES RECEIVING OFFICE

PTO-1382 (Rev. 04-2003) (Modified)

PCTUS2.FRP /REV03

Date

C-574

International Application

Attorney Docket No.

93

I. Certification under 37 CFR 1.10 (if applicable)

EL615313267
Express Mail mailing number

November 5, 2004
Date of Deposit

I hereby certify that the application/correspondence attached hereto is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

<i>Nancy Stedler</i>
Signature of person mailing correspondence

Nancy Stedler
Typed or printed name of person mailing correspondence

II. ☒ New International Application

TITLE	WATER SOLUBLE ENERGY CURABLE STEREO-CROSSLINKABLE IONOMER COMPOSITIONS
-------	--

Earliest priority date (Day/Month/Year)
06/11/03

SCREENING DISCLOSURE INFORMATION: In order to assist in screening the accompanying international application for purposes of determining whether a license for foreign transmittal should and could be granted and for other purposes, the following information is supplied. (Note: check as many boxes as apply):

- A. ☐ The invention disclosed was **not** made in the United States.
- B. ☐ There is no prior U.S. application relating to this invention.
- C. ☒ The following prior U.S. application(s) contain subject matter which is related to the invention disclosed in the attached international application. (NOTE: priority to these applications may or may not be claimed on form PCT/RO/101 (Request) and this listing does not constitute a claim for priority).

application no.	10/702,313	filed on	06/11/03
application no.		filed on	

- D. ☐ The present international application contains additional subject matter not found in the prior U.S. application(s) identified in paragraph C. above. The additional subject matter is found on pages and ☐ DOES NOT ALTER ☐ MIGHT BE CONSIDERED TO ALTER the general nature of the invention in a manner which would require the U.S. application to have been made available for inspection by the appropriate defense agencies under 35 U.S.C. 181 and 37 CFR 5.1. See 37 CFR 5.15.

III. ☐ A Response to an Invitation from the RO/US. The following document(s) is (are) enclosed:

- A. ☐ A Request for An Extension of Time to File a Response
- B. ☐ A Power of Attorney (General or Regular)
- C. ☐ Replacement pages:

pages		of the request (PCT/RO/101)	pages		of the figures
pages		of the description	pages		of the abstract
pages		of the claims			

- D. ☐ Submission of Priority Documents

Priority document		Priority document	
-------------------	--	-------------------	--

- E. ☐ Fees as specified on attached Fee Calculation sheet form PCT/RO/101 annex

IV. ☐ A Request for Rectification under PCT 91 ☐ A Petition ☐ A Sequence Listing Diskette

V. ☐ Other (please specify):

The person signing this form is the:

☐ Applicant	Sidney Persley
☒ Attorney/Agent (Reg. No.) 34,998	Typed name of signer
☐ Common Representative	<i>Sidney Persley</i> Signature

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference (if desired) (12 characters maximum) C-574

Box No. I TITLE OF INVENTION

WATER SOLUBLE ENERGY CURABLE STEREO-CROSSLINKABLE IONOMER COMPOSITIONS

Box No. II APPLICANT

☐ This person is also inventor

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

SUN CHEMICAL CORPORATION
222 Bridge Plaza South
Fort Lee, New Jersey 07024
United States of America

Telephone No.

(201) 224-4600

Facsimile No.

(201) 224-2439

Teleprinter No.

Applicant's registration No. with the

State (that is, country) of nationality:

US

State (that is, country) of residence:

US

This person is applicant for the purposes of:

☐ all designated States

☒ all designated States except the United States of

☐ the United States of America only

☐ the States indicated in the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

LINZER, Linzer
417 Seventh Street
Carlstadt, NJ 07072

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only (If this check-box is marked, do not fill in below.)

Applicant's registration No. with the Office

State (that is, country) of nationality:

US

State (that is, country) of residence:

US

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of

☒ the United States of America only

☐ the States indicated in the Supplemental Box

☒ Further applicants and/or (further) inventors are indicated on a continuation sheet.

Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as:

☒ agent

☐ common representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

PERSLEY, Sidney
222 Bridge Plaza South
Fort Lee, New Jersey 07024
United States of America

Telephone No.

(201) 224-4600

Facsimile No.

(201) 224-2439

Teleprinter No.

Agent's registration No. with the Office

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

If none of the following sub-boxes is used, this sheet should not be included in the request.

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

JONES, Richard M.
161 Greendale Drive
Oak Ridge, New Jersey 07438
United States of America

This person is:

- ☐ applicant only
☒ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

Applicant's registration No. with the

State (that is, country) of nationality:
US

State (that is, country) of residence:
US

This person is
applicant

- ☐ all designated States ☐ all designated States except the United States of ☒ the United States of America only ☐ the States indicated in the Supplemental

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
☐ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

Applicant's registration No. with the Office

State (that is, country) of nationality:

State (that is, country) of residence:

This person is
applicant for the

- ☐ all designated States ☐ all designated States except the United States of ☐ the United States of America only ☐ the States indicated in the Supplemental

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
☐ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

Applicant's registration No. with the

State (that is, country) of nationality:

State (that is, country) of residence:

This person is
applicant for the

- ☐ all designated States ☐ all designated States except the United States of ☐ the United States of America only ☐ the States indicated in the Supplemental

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
☐ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

Applicant's registration No. with the

State (that is, country) of nationality:

State (that is, country) of residence:

This person is
applicant for the

- ☐ all designated States ☐ all designated States except the United States of ☐ the United States of America only ☐ the States indicated in the Supplemental

☐ Further applicants and/or (further) inventors are indicated on another continuation sheet.

9.6

Box No. V DESIGNATIONS

The filing of this request constitutes under Rule 4.9(a), the designation of all Contracting States bound by the PCT on the international filing date, for the grant of every kind of protection available and, where applicable, for the grant of both regional and national patents.

However,

- ☐ DE Germany is not designated for any kind of national protection
- ☐ KR Republic of Korea is not designated for any kind of national protection
- ☐ RU Russian Federation is not designated for any kind of national protection

(The check-boxes above may be used to exclude (irrevocably) the designations concerned in order to avoid the ceasing of the effect, under the national law, of an earlier national application from which priority is claimed. See the Notes to Box No. V as to the consequences of such national law provisions in these and certain other States.)

Box No. VI PRIORITY CLAIM

The priority of the following earlier application(s) is hereby claimed:

Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country or Member	regional application:* regional Office	international application: receiving Office
item (1) 06/11/03	10/702,313	US		
item (2)				
item (3)				

☐ Further priority claims are indicated in the Supplemental Box.

The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of this international application is the receiving Office) identified above as:

☐ all items ☒ item (1) ☐ item (2) ☐ item (3) ☐ other, see Supplemental Box

* Where the earlier application is an ARIPO application, indicate at least one country party to the Paris Convention for the Protection of Industrial Property or one Member of the World Trade Organization for which that earlier application was filed (Rule 4.10(b)(ii)):

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):

ISA/ EPO

Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority):

Date (day/month/year) Number Country (or regional Office)

Box No. VIII DECLARATIONS

The following declarations are contained in Boxes Nos. VIII (i) to (v) (mark the applicable check-boxes below and indicate in the right column the number of each type of declaration):

		Number of declarations
☐ Box No. VIII (i)	Declaration as to the identity of the inventor	:
☐ Box No. VIII (ii)	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	:
☐ Box No. VIII (iii)	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	:
☐ Box No. VIII (iv)	Declaration of inventorship (only for the purposes of the designation of the United States of America)	:
☐ Box No. VIII (v)	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty:	:

Box No. IX CHECK LIST; LANGUAGE OF FILING

This international application contains:

(a) in **paper form**, the following number of sheets:

request (including declaration sheets) : 4
description (excluding sequence listing and/or tables related thereto) : 13
claims : 3
abstract : 1
drawings : _____

Sub-total number of sheets 21

sequence listing :
tables related thereto :
(for both, actual number of sheets if filed in paper form, whether or not also filed in computer readable form; see (c)) _____

Total number of sheets : 21

(b) ☐ **only in computer readable form** (Section 801(a)(i))

(i) ☐ sequence listing
(ii) ☐ tables related thereto

(c) ☐ **also in computer readable form** (Section 801(a)(ii))

(i) ☐ sequence listing
(ii) ☐ tables related thereto

Type and number of carriers
(diskette, CD-ROM, CD-R or other)
on which are contained the

☐ sequence listing:
☐ tables related thereto:
(additional copies to be indicated under items 9(ii) and/or 10(ii), in right column)

This international application is **accompanied** by the following item(s) (mark the applicable check-boxes below and indicate in right column the number of each item):

Number of items

1. ☒ fee calculation sheet
2. ☐ original separate power of attorney
3. ☐ original general power of attorney
4. ☐ copy of general power of attorney; reference number, if any: _____
5. ☐ statement explaining lack of signature
6. ☐ priority document(s) identified in Box No. VI as item(s): _____
7. ☐ translation of international application into (language): _____
8. ☐ separate indications concerning deposited microorganism or other biological material
9. ☐ sequence listing in computer readable form (indicate type and number of carriers)
 - (i) ☐ copy submitted for the purposes of international search under Rule 13ter only (and not as part of the international application)
 - (ii) ☐ (only where check-box (b)(i) or (c)(i) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Rule 13ter
 - (iii) ☐ together with relevant statement as to the identity of the copy or copies with the sequence listing mentioned in left column
10. ☐ tables in computer readable form related to sequence listing (indicate type and number of carriers)
 - (i) ☐ copy submitted for the purposes of international search under Section 802(b-quater) only (and not as part of the international application)
 - (ii) ☐ (only where check-box (b)(ii) or (c)(ii) is marked in left column) additional copies including, where applicable, the copy for the purposes of international search under Section 802(b-quater)
 - (iii) ☐ together with relevant statement as to the identity of the copy or copies with the tables mentioned in left column
11. ☐ other (specify): _____

Figure of the drawings which should accompany the abstract:

Language of filing of the international application: English

Box No. X SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

Volker Linzer

Richard M. Jones

For receiving Office use only		2. Drawings: ☐ received: ☐ not received:
1. Date of actual receipt of the purported international application:		
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:		
4. Date of timely receipt of the required corrections under PCT Article 11(2):		
5. International Searching Authority (if two or more are competent): ISA/	6. ☐ Transmittal of search copy delayed until search fee is paid	

For International Bureau use only

Date of receipt of the record copy by the International Bureau:

98

PCT

FEE CALCULATION SHEET

Annex to the Request

For receiving Office use only

International Application No.

Date stamp of the receiving Office

Applicant's or agent's
file reference

C-574

Applicant

SUN CHEMICAL CORPORATION

CALCULATION OF PRESCRIBED FEES

1. TRANSMITTAL FEE 300.00 **T**

2. SEARCH FEE 1,920.00 **S**

International search to be carried out by EPO
(If two or more International Searching Authorities are competent to carry out the international search, indicate the name of the Authority which is chosen to carry out the international search.)

3. INTERNATIONAL FILING FEE

Where item (b) and/or (c) of Box No. IX apply, enter Sub-total number of sheets } 19
Where item (b) and (c) of Box No. IX do not apply, enter Total number of sheets }

i1 first 30 sheets 1,053.00 **i1**

i2 0 x = 0.00 **i2**
number of sheets in excess of 30 fee per sheet

i3 additional component (only if sequence listing and/or tables related thereto are filed in computer readable form under Section 801(a)(i), or both in that form and on paper, under Section 801(a)(ii)):

400 x = **i3**
fee per sheet

Add amounts entered at i1, i2 and i3 and enter total at I 1,053.00 **I**

(Applicants from certain States are entitled to a reduction of 75% of the international filing fee. Where the applicant is (or all applicants are) so entitled,

4. FEE FOR PRIORITY DOCUMENT (if applicable) 20.00 **P**

5. TOTAL FEES PAYABLE 3,293.00

Add amounts entered at T, S, I and P, and enter total in the TOTAL box

TOTAL

MODE OF PAYMENT

- ☒ authorization to charge deposit account (see below) ☐ postal money order ☐ cash ☐ coupons
☐ cheque ☐ bank draft ☐ revenue stamps ☐ other (specify):

AUTHORIZATION TO CHARGE (OR CREDIT) DEPOSIT ACCOUNT

(This mode of payment may not be available at all receiving Offices)

- ☒ Authorization to charge the total fees indicated above.
☒ (This check-box may be marked only if the conditions for deposit accounts of the receiving Office so permit) Authorization to charge any deficiency or credit any overpayment in the total fees indicated above.
☒ Authorization to charge the fee for priority document.

Receiving Office: US

Deposit Account No.: 19-4968

Date: November 5, 2004

Name: Sidney Persley

Signature: Sidney Persley

From the INTERNATIONAL BUREAU

PCT

**SECOND AND SUPPLEMENTARY NOTICE
INFORMING THE APPLICANT OF THE
COMMUNICATION OF THE INTERNATIONAL
APPLICATION (TO DESIGNATED OFFICES
WHICH APPLY THE 30 MONTH TIME
LIMIT UNDER ARTICLE 22(1))**

(PCT Rule 47.1(c))

To:

PERSLEY, Sidney
c/o Sun Chemical Corporation
35 Waterview Blvd.
Parsippany, NJ 07054
ETATS-UNIS D'AMERIQUE

RECEIVED
MAR 20 2006
SIDNEY PERSLEY

Date of mailing (day/month/year)

09 March 2006 (09.03.2006)

Applicant's or agent's file reference

C-574

IMPORTANT NOTICE

International application No.

PCT/US2004/037222

International filing date (day/month/year)

05 November 2004 (05.11.2004)

Priority date (day/month/year)

06 November 2003 (06.11.2003)

Applicant

SUN CHEMICAL CORPORATION et al

1. **ATTENTION:** For any designated Office(s), for which the time limit under Article 22(1), as in force from 1 April 2002 (30 months from the priority date), **does not apply**, please see Form PCT/IB/308(First Notice) issued previously.

2. Notice is hereby given that the following designated Office(s), for which the time limit under Article 22(1), as in force from 1 April 2002, **does apply**, has/have requested that the communication of the international application, as provided for in Article 20, be effected under Rule 93bis.1. The International Bureau has effected that communication on the date indicated below:
26 May 2005 (26.05.2005)

AU, AZ, BY, CN, CO, DZ, EP, HU, KG, KP, KR, MD, MK, MZ, NA, PG, RU, SY, TM, US

In accordance with Rule 47.1(c-bis)(i), those Offices will accept the present notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

3. The following designated Offices, for which the time limit under Article 22(1), as in force from 1 April 2002, **does apply**, have not requested, as at the time of mailing of the present notice, that the communication of the international application be effected under Rule 93bis.1:

AE, AG, AL, AM, AP, AT, BA, BB, BG, BR, BW, BZ, CA, CR, CU, CZ, DE, DK, DM, EA, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, ID, IL, IN, IS, JP, KE, KZ, LC, LK, LR, LS, LT, LV, MA, MG, MN, MW, MX, NI, NO, NZ, OA, OM, PH, PL, PT, RO, SC, SD, SG, SK, SL, TJ, TN, TR, TT, UA, UZ, VC, VN, YU, ZA, ZW

In accordance with Rule 47.1(c-bis)(ii), those Offices accept the present notice as conclusive evidence that the Contracting State for which that Office acts as a designated Office does not require the furnishing, under Article 22, by the applicant of a copy of the international application.

4. TIME LIMITS for entry into the national phase

For the designated or elected Office(s) listed above, the applicable time limit for entering the national phase will, **subject to what is said in the following paragraph**, be **30 MONTHS** from the priority date.

In practice, **time limits other than the 30-month time limit** will continue to apply, for various periods of time, in respect of certain of the designated or elected Office(s) listed above. For **regular updates on the applicable time limits** (30 or 31 months, or other time limit), Office by Office, refer to the *PCT Gazette*, the *PCT Newsletter* and the *PCT Applicant's Guide*, Volume II, National Chapters, all available from WIPO's Internet site, at <http://www.wipo.int/pct/en/index.html>.

It is the applicant's **sole responsibility** to monitor all these time limits.

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Authorized officer

Simin Baharlou

Facsimile No.+41 22 740 14 35

Facsimile No.+41 22 338 71 30

PCT

NOTIFICATION CONCERNING
SUBMISSION OR TRANSMITTAL
OF PRIORITY DOCUMENT

(PCT Administrative Instructions, Section 411)

From the INTERNATIONAL BUREAU

To:

PERSLEY, Sidney
222 Bridge Plaza South
Fort Lee, NJ 07024
ETATS-UNIS D'AMERIQUE

Date of mailing (day/month/year) 17 January 2005 (17.01.2005)	RECEIVED JAN 27 2005 SIDNEY PERSLEY	IMPORTANT NOTIFICATION	
Applicant's or agent's file reference C-574		International application No. PCT/US04/037222	International filing date (day/month/year) 05 November 2004 (05.11.2004)
International publication date (day/month/year)		Priority date (day/month/year) 06 November 2003 (06.11.2003)	
Applicant SUN CHEMICAL CORPORATION et al			

- By means of this Form, which replaces any previously issued notification concerning submission or transmittal of priority documents, the applicant is hereby notified of the date of receipt by the International Bureau of the priority document(s) relating to all earlier application(s) whose priority is claimed. Unless otherwise indicated by the letters "NR", in the right-hand column or by an asterisk appearing next to a date of receipt, the priority document concerned was submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b).
- (If applicable)* The letters "NR" appearing in the right-hand column denote a priority document which, on the date of mailing of this Form, had not yet been received by the International Bureau under Rule 17.1(a) or (b). Where, under Rule 17.1(a), the priority document must be submitted by the applicant to the receiving Office or the International Bureau, but the applicant fails to submit the priority document within the applicable time limit under that Rule, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.
- (If applicable)* An asterisk (*) appearing next to a date of receipt, in the right-hand column, denotes a priority document submitted or transmitted to the International Bureau but not in compliance with Rule 17.1(a) or (b) (the priority document was received after the time limit prescribed in Rule 17.1(a) or the request to prepare and transmit the priority document was submitted to the receiving Office after the applicable time limit under Rule 17.1(b)). Even though the priority document was not furnished in compliance with Rule 17.1(a) or (b), the International Bureau will nevertheless transmit a copy of the document to the designated Offices, for their consideration. In case such a copy is not accepted by the designated Office as the priority document, Rule 17.1(c) provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.

Priority date	Priority application No.	Country or regional Office or PCT receiving Office	Date of receipt of priority document
06 November 2003 (06.11.2003)	10/702,313	US	20 December 2004 (20.12.2004)

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Authorized officer

Olaiz Alicia

Facsimile No. +41 22 740 14 35

Facsimile No. +41 22 338 71 30

Telephone No. +41 22 338 9288

PATENT COOPERATION TREATY

PCT

From the INTERNATIONAL BUREAU

NOTIFICATION OF RECEIPT OF
RECORD COPY

(PCT Rule 24.2(a))

To:

PERSLEY, Sidney
222 Bridge Plaza South
Fort Lee, NJ 07024
United States of America

RECEIVED
JAN 18 2005
SIDNEY PERSLEY

Date of mailing (day/month/year) 14 January 2005 (14.01.2005)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference C-574	International application No. PCT/US2004/037222

The applicant is hereby notified that the International Bureau has received the record copy of the international application as detailed below.

Name(s) of the applicant(s) and State(s) for which they are applicants:

SUN CHEMICAL CORPORATION (for all designated States except US)

LINZER, Linzer et al (for US)

International filing date : 05 November 2004 (05.11.2004)

Priority date(s) claimed : 06 November 2003 (06.11.2003)

Date of receipt of the record copy
by the International Bureau : 03 January 2005 (03.01.2005)

List of designated Offices :

AP : BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW

EA : AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

EP : AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR

OA : BF, BJ, CF, CG, CI, CM, GA, GN, GO, GW, ML, MR, NE, SN, TD, TG

National : AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Facsimile No. (41-22) 338.71.30

Authorized officer:

R. Meach (Fax 338-71-30)

Telephone No. (41-22) 338 9266

Continuation of Form PCT/IB/301

NOTIFICATION OF RECEIPT OF RECORD COPY

102

Date of mailing (day/month/year) 14 January 2005 (14.01.2005)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference C-574	International application No. PCT/US2004/037222

ATTENTION

The applicant should carefully check the data appearing in this Notification. In case of any discrepancy between these data and the indications in the international application, the applicant should immediately inform the International Bureau.

In addition, the applicant's attention is drawn to the information contained in the Annex, relating to:

☒ time limits for entry into the national phase - see updated important information (as of April 2002)

☒ requirements regarding priority documents (if applicable)

A copy of this Notification is being sent to the receiving Office and to the International Searching Authority.

INFORMATION ON TIME LIMITS FOR ENTERING THE NATIONAL PHASE 103

The applicant is reminded that the **"national phase" must be entered** before each of the designated Offices indicated on the cover sheet of this Notification by paying national fees and furnishing translations, as prescribed by Articles 22 and 39 and the applicable national laws. In addition, the applicant may also have to comply **with other special requirements** applicable in certain Offices. It is the **applicant's responsibility** to ensure the necessary steps to enter the national phase are taken in a timely fashion. Most Offices do not issue reminders to applicants in connection with the entry into the national phase.

The **applicable time limit** for entering the national phase will, **subject to what is said in the following paragraph**, be **30 MONTHS** from the priority date, not only in respect of any elected Office if a demand for international preliminary examination is filed before the expiration of 19 months from the priority date (see Article 39(1)), but also in respect of any designated Office, in the absence of filing of such demand, where Article 22(1) as modified with effect from 1 April 2002 applies in respect of that designated Office. For further details, see PCT Gazette No. 44/2001 of 1 November 2001, pages 19926, 19932 and 19934, as well as the PCT Newsletter, October and November 2001 and February 2002 issues.

In practice, **time limits other than the 30-month time limit will continue to apply, for various periods of time**, in respect of certain designated or elected Offices. For **regular updates on the applicable time limits (20, 21, 30 or 31 months, or other time limit)**, Office by Office, refer to the PCT Gazette ("Section IV" part published on a weekly basis), to the PCT Newsletter (on a monthly basis) and to the relevant National Chapters in Volume II of the PCT Applicant's Guide (the paper version of which is updated usually twice a year and the Internet version of which is updated usually on a weekly basis). Finally, a cumulative table of all applicable time limits for entering the national phase is available from WIPO's Internet site, via links from various pages the site including those of the Gazette, Newsletter and Guide, at <http://www.wipo.int/pct/en/index.html>.

Information about the requirements for **filing a demand for international preliminary examination** is set out in the PCT Applicant's Guide, Volume I/A, Chapter IX. Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination (at present, all PCT Contracting States are bound by Chapter II).

REQUIREMENTS REGARDING PRIORITY DOCUMENTS

For applicants who have not yet complied with the requirements regarding priority documents, the following is recalled.

Where the priority of an earlier national, regional or international application is claimed, the applicant must submit a copy of the said earlier application, certified by the authority with which it was filed ("the priority document") to the receiving Office (which will transmit it to the International Bureau) or directly to the International Bureau, before the expiration of 16 months from the priority date, provided that any such priority document may still be submitted to the International Bureau before that date of international publication of the international application, in which case that document will be considered to have been received by the International Bureau on the last day of the 16-month time limit (Rule 17.1(a)).

Where the priority document is issued by the receiving Office, the applicant may, instead of submitting the priority document, request the receiving Office to prepare and transmit the priority document to the International Bureau. Such request must be made before the expiration of the 16-month time limit and may be subjected by the receiving Office to the payment of a fee (Rule 17.1(b)).

If the priority document concerned is not submitted to the International Bureau or if the request to the receiving Office to prepare and transmit the priority document has not been made (and the corresponding fee, if any, paid) within the applicable time limit indicated under the preceding paragraphs, any designated State may disregard the priority claim, provided that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within the time limit which is reasonable under the circumstances (Rule 17.1(c)).

Where several priorities are claimed, the priority date to be considered for the purposes of computing the 16-month time limit (and all other PCT time limits) is the filing date of the earliest application whose priority is claimed (Article 2(xi)(b)).

PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

104

To:
SUN CHEMICAL CORPORATION
Attn. Persley, Sidney
222 Bridge Plaza South
Fort Lee, NJ 07024
UNITED STATES OF AMERICA

**NOTIFICATION OF RECEIPT
OF SEARCH COPY**

RECEIVED (PCT Rule 25.1)

FEB 07 2005

SIDNEY PEARLEY

Date of mailing (day/month/year)	27/01/2005
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Applicant's or agent's file reference
C-574

IMPORTANT NOTIFICATION

International application No. PCT/US2004/037222	International filing date(day/month/year) 05/11/2004	Priority date (day/month/year) 06/11/2003
---	--	---

Applicant
SUN CHEMICAL CORPORATION

1. **Where the International Searching Authority and the receiving Office are not the same office:**

The applicant is hereby notified that the search copy of the international application was received by this International Searching Authority on the date indicated below.

Where the International Searching Authority and the receiving Office are the same office:

The applicant is hereby notified that the search copy of the international application was received on the date indicated below.

31/12/2004 (date of receipt).

2. ☐ The search copy was accompanied by a nucleotide and/or amino acid sequence listing or tables related thereto in computer readable form.

3. **Time limit for establishment of international search report and written opinion of the International Searching Authority**

The applicant is informed that the time limit for establishing the international search report and the written opinion of the international Searching Authority is three months from the date of receipt indicated above or nine months from the priority date, whichever time limit expires later (Rules 42.1 and 43bis.1(a))

4. A copy of this notification has been sent to the International Bureau and, where the first sentence of paragraph 1 applies, to the receiving Office.

Name and mailing address of the International Searching Authority  European Patent Office, P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer ISA/EP
--	---

PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

To:
SUN CHEMICAL CORPORATION
Attn. Persley, Sidney
222 Bridge Plaza South
Fort Lee, NJ 07024
UNITED STATES OF AMERICA

RECEIVED

MAR 28 2005

SIDNEY PERSLEY

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL SEARCH REPORT AND
THE WRITTEN OPINION OF THE INTERNATIONAL
SEARCHING AUTHORITY, OR THE DECLARATION

(PCT Rule 44.1)

Applicant's or agent's file reference

C-574

Date of mailing
(day/month/year)

24/03/2005

FOR FURTHER ACTION

See paragraphs 1 and 4 below

International application No.

PCT/US2004/037222

International filing date
(day/month/year)

05/11/2004

Applicant

SUN CHEMICAL CORPORATION

1. ☒ The applicant is hereby notified that the international search report and the written opinion of the International Searching Authority have been established and are transmitted herewith.

Filing of amendments and statement under Article 19:

The applicant is entitled, if he so wishes, to amend the claims of the International Application (see Rule 46):

When? The time limit for filing such amendments is normally 2 months from the date of transmittal of the International Search Report; however, for more details, see the notes on the accompanying sheet.

Where? Directly to the International Bureau of WIPO, 34 chemin des Colombettes
1211 Geneva 20, Switzerland, Facsimile No.: (41-22) 740.14.35

For more detailed instructions, see the notes on the accompanying sheet.

2. ☐ The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect and the written opinion of the International Searching Authority are transmitted herewith.

3. ☐ **With regard to the protest** against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

- ☐ the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.
☐ no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.

4. Reminders

Shortly after the expiration of **18 months** from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in Rules 90bis.1 and 90bis.3, respectively, before the completion of the technical preparations for international publication.

The applicant may submit comments on an informal basis on the written opinion of the International Searching Authority to the International Bureau. The International Bureau will send a copy of such comments to all designated Offices unless an international preliminary examination report has been or is to be established. These comments would also be made available to the public but not before the expiration of 30 months from the priority date.

Within **19 months** from the priority date, but only in respect of some designated Offices, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until **30 months** from the priority date (in some Offices even later); otherwise, the applicant must, **within 20 months** from the priority date, perform the prescribed acts for entry into the national phase before those designated Offices.

In respect of other designated Offices, the time limit of **30 months** (or later) will apply even if no demand is filed within 19 months.

See the Annex to Form PCT/IB/301 and, for details about the applicable time limits, Office by Office, see the *PCT Applicant's Guide*, Volume II, National Chapters and the WIPO Internet site.

Name and mailing address of the International Searching Authority



European Patent Office, P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Monika Schmitz

NOTES TO FORM PCT/ISA/220

These Notes are intended to give the basic instructions concerning the filing of amendments under article 19. The Notes are based on the requirements of the Patent Cooperation Treaty, the Regulations and the Administrative Instructions under that Treaty. In case of discrepancy between these Notes and those requirements, the latter are applicable. For more detailed information, see also the PCT Applicant's Guide, a publication of WIPO.

In these Notes, "Article", "Rule", and "Section" refer to the provisions of the PCT, the PCT Regulations and the PCT Administrative Instructions respectively.

INSTRUCTIONS CONCERNING AMENDMENTS UNDER ARTICLE 19

The applicant has, after having received the international search report, one opportunity to amend the claims of the international application. It should however be emphasized that, since all parts of the international application (claims, description and drawings) may be amended during the international preliminary examination procedure, there is usually no need to file amendments of the claims under Article 19 except where, e.g. the applicant wants the latter to be published for the purposes of provisional protection or has another reason for amending the claims before international publication. Furthermore, it should be emphasized that provisional protection is available in some States only.

What parts of the international application may be amended?

Under Article 19, only the claims may be amended.

During the international phase, the claims may also be amended (or further amended) under Article 34 before the International Preliminary Examining Authority. The description and drawings may only be amended under Article 34 before the International Examining Authority.

Upon entry into the national phase, all parts of the international application may be amended under Article 28 or, where applicable, Article 41.

When?

Within 2 months from the date of transmittal of the international search report or 16 months from the priority date, whichever time limit expires later. It should be noted, however, that the amendments will be considered as having been received on time if they are received by the International Bureau after the expiration of the applicable time limit but before the completion of the technical preparations for international publication (Rule 46.1).

Where not to file the amendments?

The amendments may only be filed with the International Bureau and not with the receiving Office or the International Searching Authority (Rule 46.2).

Where a demand for international preliminary examination has been/is filed, see below.

How?

Either by cancelling one or more entire claims, by adding one or more new claims or by amending the text of one or more of the claims as filed.

A replacement sheet must be submitted for each sheet of the claims which, on account of an amendment or amendments, differs from the sheet originally filed.

All the claims appearing on a replacement sheet must be numbered in Arabic numerals. Where a claim is cancelled, no renumbering of the other claims is required. In all cases where claims are renumbered, they must be renumbered consecutively (Administrative Instructions, Section 205(b)).

The amendments must be made in the language in which the international application is to be published.

What documents must/may accompany the amendments?

Letter (Section 205(b)):

The amendments must be submitted with a letter.

The letter will not be published with the international application and the amended claims. It should not be confused with the "Statement under Article 19(1)" (see below, under "Statement under Article 19(1)").

The letter must be in English or French, at the choice of the applicant. However, if the language of the international application is English, the letter must be in English; if the language of the international application is French, the letter must be in French.

NOTES TO FORM PCT/ISA/220 (continued)

108

The letter must indicate the differences between the claims as filed and the claims as amended. It must, in particular, indicate, in connection with each claim appearing in the international application (it being understood that identical indications concerning several claims may be grouped), whether

- (i) the claim is unchanged;
- (ii) the claim is cancelled;
- (iii) the claim is new;
- (iv) the claim replaces one or more claims as filed;
- (v) the claim is the result of the division of a claim as filed.

The following examples illustrate the manner in which amendments must be explained in the accompanying letter:

1. [Where originally there were 48 claims and after amendment of some claims there are 51]:
"Claims 1 to 29, 31, 32, 34, 35, 37 to 48 replaced by amended claims bearing the same numbers; claims 30, 33 and 36 unchanged; new claims 49 to 51 added."
2. [Where originally there were 15 claims and after amendment of all claims there are 11]:
"Claims 1 to 15 replaced by amended claims 1 to 11."
3. [Where originally there were 14 claims and the amendments consist in cancelling some claims and in adding new claims]:
"Claims 1 to 6 and 14 unchanged; claims 7 to 13 cancelled; new claims 15, 16 and 17 added." or
"Claims 7 to 13 cancelled; new claims 15, 16 and 17 added; all other claims unchanged."
4. [Where various kinds of amendments are made]:
"Claims 1-10 unchanged; claims 11 to 13, 18 and 19 cancelled; claims 14, 15 and 16 replaced by amended claim 14; claim 17 subdivided into amended claims 15, 16 and 17; new claims 20 and 21 added."

"Statement under article 19(1)" (Rule 46.4)

The amendments may be accompanied by a statement explaining the amendments and indicating any impact that such amendments might have on the description and the drawings (which cannot be amended under Article 19(1)).

The statement will be published with the international application and the amended claims.

It must be in the language in which the international application is to be published.

It must be brief, not exceeding 500 words if in English or if translated into English.

It should not be confused with and does not replace the letter indicating the differences between the claims as filed and as amended. It must be filed on a separate sheet and must be identified as such by a heading, preferably by using the words "Statement under Article 19(1)."

It may not contain any disparaging comments on the international search report or the relevance of citations contained in that report. Reference to citations, relevant to a given claim, contained in the international search report may be made only in connection with an amendment of that claim.

Consequence if a demand for international preliminary examination has already been filed

If, at the time of filing any amendments under Article 19, a demand for international preliminary examination has already been submitted, the applicant must preferably, at the same time of filing the amendments with the International Bureau, also file a copy of such amendments with the International Preliminary Examining Authority (see Rule 62.2(a), first sentence).

Consequence with regard to translation of the international application for entry into the national phase

The applicant's attention is drawn to the fact that, where upon entry into the national phase, a translation of the claims as amended under Article 19 may have to be furnished to the designated/elected Offices, instead of, or in addition to, the translation of the claims as filed.

For further details on the requirements of each designated/elected Office, see Volume II of the PCT Applicant's Guide.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference C-574	FOR FURTHER ACTION see Form PCT/ISA/220 as well as, where applicable, item 5 below.	
International application No. PCT/US2004/037222	International filing date (day/month/year) 05/11/2004	(Earliest) Priority Date (day/month/year) 06/11/2003
Applicant SUN CHEMICAL CORPORATION		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 4 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ The international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

- b. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, see Box No. I.

2. ☐ **Certain claims were found unsearchable** (See Box II).

3. ☐ **Unity of invention is lacking** (see Box III).

4. With regard to the title,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the abstract,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. With regard to the drawings,

- a. the figure of the **drawings** to be published with the abstract is Figure No. _____

☐ as suggested by the applicant.

☐ as selected by this Authority, because the applicant failed to suggest a figure.

☐ as selected by this Authority, because this figure better characterizes the invention.

- b. ☐ none of the figures is to be published with the abstract.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US2004/037222

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C08F8/00 C08F8/44

109

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 7 C08F C09D

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)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 559 222 B1 (ROONEY JOHN ET AL) 6 May 2003 (2003-05-06) cited in the application the whole document	1-8, 10, 11, 13, 14, 16
X	US 4 745 138 A (THANAWALLA ET AL) 17 May 1988 (1988-05-17) cited in the application the whole document	1, 4-8, 11, 14
X	US 3 425 977 A (MARTIN SKOULTCHI ET AL) 4 February 1969 (1969-02-04) the whole document	1, 4-8, 11, 14
X	US 3 862 067 A (MOTIER ET AL) 21 January 1975 (1975-01-21) the whole document	1, 4-8, 11, 14
	----- -/--	



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

G document member of the same patent family

Date of the actual completion of the international search

10 March 2005

Date of mailing of the international search report

24/03/2005

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040. Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Droghetti, A

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US2004/037222

110

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 633 307 A (DAS ET AL) 27 May 1997 (1997-05-27) the whole document -----	9,12,15

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US2004/037222

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 6559222	B1	06-05-2003	CA 2306478 A1 22-04-1999 EP 1023360 A2 02-08-2000 WO 9919369 A2 22-04-1999
US 4745138	A	17-05-1988	NONE
US 3425977	A	04-02-1969	NONE
US 3862067	A	21-01-1975	BE 798169 A1 15-10-1973 DE 2318555 A1 18-10-1973 FR 2180073 A1 23-11-1973 GB 1413001 A 05-11-1975 IT 983008 B 31-10-1974 JP 49018124 A 18-02-1974 NL 7304875 A 16-10-1973 SE 380285 B 03-11-1975
US 5633307	A	27-05-1997	AT 178918 T 15-04-1999 AU 3640495 A 03-07-1996 CA 2204178 A1 20-06-1996 DE 69509119 D1 20-05-1999 DE 69509119 T2 23-12-1999 EP 0797607 A1 01-10-1997 ES 2133808 T3 16-09-1999 JP 10502700 T 10-03-1998 WO 9618668 A1 20-06-1996 US 6090881 A 18-07-2000

PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

PCT

To:

see form PCT/ISA/220

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY (PCT Rule 43bis.1)

Date of mailing
(day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference
see form PCT/ISA/220

FOR FURTHER ACTION
See paragraph 2 below

International application No.
PCT/US2004/037222

International filing date (day/month/year)
05.11.2004

Priority date (day/month/year)
06.11.2003

International Patent Classification (IPC) or both national classification and IPC
C08F8/00, C08F8/44

Applicant
SUN CHEMICAL CORPORATION

1. This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☒ Box No. VII Certain defects in the international application
- ☒ Box No. VIII Certain observations on the international application

2. FURTHER ACTION

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA"). However, this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1bis(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of three months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

3. For further details, see notes to Form PCT/ISA/220.

Name and mailing address of the ISA:



European Patent Office - P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk - Pays Bas
Tel. +31 70 340 - 2040 Tx: 31 651 epo nl
Fax: +31 70 340 - 3016

Authorized Officer

Droghetti, A

Telephone No. +31 70 340-4143



**WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING AUTHORITY**

113
International application No.
PCT/US2004/037222

Box No. I Basis of the opinion

1. With regard to the **language**, this opinion has been established on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
 - ☐ This opinion has been established on the basis of a translation from the original language into the following language , which is the language of a translation furnished for the purposes of international search (under Rules 12.3 and 23.1(b)).
2. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application and necessary to the claimed invention, this opinion has been established on the basis of:
 - a. type of material:
 - ☐ a sequence listing
 - ☐ table(s) related to the sequence listing
 - b. format of material:
 - ☐ in written format
 - ☐ in computer readable form
 - c. time of filing/furnishing:
 - ☐ contained in the international application as filed.
 - ☐ filed together with the international application in computer readable form.
 - ☐ furnished subsequently to this Authority for the purposes of search.
3. ☐ In addition, in the case that more than one version or copy of a sequence listing and/or table relating thereto has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.
4. Additional comments:

**WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING AUTHORITY**

114
International application No.
PCT/US2004/037222

Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	
	No: Claims	1-16
Inventive step (IS)	Yes: Claims	
	No: Claims	1-16
Industrial applicability (IA)	Yes: Claims	1-16
	No: Claims	

2. Citations and explanations

see separate sheet

Box No. VII Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

**WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING
AUTHORITY (SEPARATE SHEET)**

115
International application No.

PCT/US2004/037222

Re Item V

Reference is made to the following document/s:

D1: US B 6559222 (cited by the applicant)
D2: US A 4745138
D3: US A 3425977
D4: US A 3862067
D5: US A 5633307

1. Novelty (Art. 33(2) PCT)

The subject-matter of claims 1-16 is not novel for the following reasons.

1.1. D1 discloses the same polymer composition for a coating composition comprising an ethylenically unsaturated acid resin, a neutralizing agent (e.g. ammonia) and water (see search report).
Note that the only part in the claims, which appears as being novel with regard to D1, is the use of an ethyl-unsaturated amine as neutralizing agent.

Thus the subject-matter of claims 1-8,10,11,13,14,16, is not novel in view of D1.

1.2. D2-D4 disclose curable coating composition comprising acid resins, a neutralizing agent (e.g. amines or ammonia) and water as claimed.

Thus the subject-matter of claims 1,4-8,11,14 is not novel in view of D2-D4

1.3. In D5 a polymer composition comprising a polyamine with acrylic functional groups, an acid and water as claimed is disclosed.

A coating composition is prepared thereof.

Note that in the present application no specific method for preparing the composition is claimed. In claims 11-14, no defined technical features characterising the process are provided.

Thus the subject-matter of claims 9,12,15 is not novel in view of D2.

2. Inventive Step (Art. 33(3) PCT)

Being not novel the subject-matter of claims 1-16 cannot be regarded as inventive either.

3. The subject-matter of claims 1-16 meets the requirements of **Article 33(4) PCT**, with regard to **industrial applicability**.

Re Item VII

Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents D1, D3-D5 is not mentioned in the description, nor are these documents identified therein.

Re Item VIII (Art. 6 PCT)

1. In claim 1 the process features regarding the curing method (see "upon curing with an actinic radiation source") do not limit the process claim rendering the same unclear.

Furthermore the wording "offering the composition an increased crosslinked density" attempts to define the invention by the result-to-be-achieved.

In cl. 1 the expression "stereo-crosslinked" has no well-known or recognized meaning in the art.

This term is vague and unclear and leaves the reader in doubt as to the meaning of the technical features to which it refers, thereby rendering the definition of the subject-matter of said claim unclear, Article 6 PCT (the same applies to the expression "energy curable" in claims 11-16).

2. Although claims 1 and 10 have been drafted as separate independent claims, they appear to relate effectively to the same subject-matter and to differ from each other only with regard to the definition of the subject-matter for which protection is sought and in respect of the terminology used for the features of that subject-matter. The aforementioned claims therefore lack conciseness and as such do not meet the requirements of Article 6 PCT.

3. Claim 9,12,15 are not supported by the description as required by Article 6 PCT, as their scope is broader than justified by the description and drawings (see page 3). No specific hint to the basic resin as claimed can be found in the description and no process or article therefrom is mentioned either.

4. Claims 1,6,10-16 are not supported by the description.

117

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
Rad: 06-054552- -00000-0000
Fec: 2006-05-05 17:19:14 Dep. 2020 NUECREACIONES
Tra. 11 PATENTE/II
Eve: 376 FASENACIONALI
Act: 114 PRESENTACION

REQUISITOS NECESARIOS PARA PRESENTACION Y AGILIZACION DEL TRAMITE

		USUARIO	RADICADOR
PATENTE DE INVENCION	MODELO DE UTILIDAD	()	()
- Indicación de que se solicita la concesión de una patente.		()	☒
- Datos de identificación del solicitante o de la persona que se presenta la solicitud.		()	☒
- Descripción de la Invención.		()	☒
- Dibujos de ser estos pertinentes.		()	☒
- Comprobante de Pago de las tasas establecidas.		()	☒
COMPLETA ()	INCOMPLETA ()	()	()
DISEÑO INDUSTRIAL ()			
- Indicación de que se solicita el registro de Diseño Industrial		()	()
- Datos de identificación del solicitante o de la persona que presenta la solicitud.		()	()
- Representación gráfica o fotográfica del Diseño Industrial o muestra del Material que incorpora el diseño.		()	()
- Comprobante de pago de las tasas establecidas		()	()
COMPLETA ()	INCOMPLETA ()	()	()
ESQUEMA DE TRAZADO ()			
- Indicación de que solicita el registro de un Esquema de trazado.		()	()
- Datos de identificación del solicitante o de la persona que presenta La solicitud.		()	()
- Representación gráfica del esquema de trazado		()	()
- Comprobante de pago de las tasas establecidas.		()	()
COMPLETA ()	INCOMPLETA ()	()	()

Firma Responsable:

Fecha: