



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification⁴ : C23G 1/00	A1	(11) International Publication Number: WO 87/ 00209 (43) International Publication Date: 15 January 1987 (15.01.87)
(21) International Application Number: PCT/US86/01361 (22) International Filing Date: 27 June 1986 (27.06.86) (31) Priority Application Number: 750,198 (32) Priority Date: 1 July 1985 (01.07.85) (33) Priority Country: US (71) Applicant: PETROFERM USA, INC. [US/US]; Amelia Island Office Park, Route 2, Box 280, Amelia Island, FL 32034 (US). (72) Inventors: HAYES, Michael, Edward ; 71 Oak Lane, Fernandina Beach, FL 32034 (US). HOOD, Craig, C. ; 3015 Club Villas, Fernandina Beach, FL 32034 (US). MILLER, Donald, E. ; 3 Beach Walker Road, Fernandina Beach, FL 32034 (US). SHARPE, Robert ; 3025 S. Fletcher Avenue, Apt. B, Fernandina Beach, FL 32034 (US).		(74) Agent: MISROCK, Leslie, S.; Pennie & Edmonds, 1155 Avenue of the Americas, New York, NY 10036 (US). (81) Designated States: AT (European patent), BE (European patent), CH (European patent), DE (European patent), FR (European patent), GB (European patent), IT (European patent), JP, LU (European patent), NL (European patent), SE (European patent). Published <i>With international search report.</i> <i>With amended claims and statement.</i>
(54) Title: COMPOSITIONS AND METHODS FOR PRINTED CIRCUIT BOARD AND/OR PRINTED WIRING BOARD CLEANING AND SOLDERMASK TESTING (57) Abstract Methods and compositions for the removal of rosin soldering flux and adhesive tape residues from printed circuit and/or wiring boards, and for testing the quality of curing of U.V.-cured soldermask on such boards. The compositions contain terpene compounds, preferably in combination with terpene emulsifying surfactants to facilitate removal by rinsing in water.		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	GA	Gabon	MR	Mauritania
AU	Australia	GB	United Kingdom	MW	Malawi
BB	Barbados	HU	Hungary	NL	Netherlands
BE	Belgium	IT	Italy	NO	Norway
BG	Bulgaria	JP	Japan	RO	Romania
BR	Brazil	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic			SE	Sweden
CG	Congo	KR	Republic of Korea	SN	Senegal
CH	Switzerland	LI	Liechtenstein	SU	Soviet Union
CM	Cameroon	LK	Sri Lanka	TD	Chad
DE	Germany, Federal Republic of	LU	Luxembourg	TG	Togo
DK	Denmark	MC	Monaco	US	United States of America
FI	Finland	MG	Madagascar		
FR	France	ML	Mali		

- 1 -

COMPOSITIONS AND METHODS FOR PRINTED CIRCUIT BOARD
AND/OR PRINTED WIRING BOARD CLEANING AND SOLDERMASK TESTING

FIELD OF THE INVENTION:

25 The present invention relates to non-toxic,
environmentally safe compositions for use in cleaning during
the fabrication of printed circuit or printed wiring boards.
Terpene compounds are utilized with or without terpene
emulsifying surfactants to achieve a variety of objectives,
among which are the removal of solder flux, oils, waxes and
greasy substances or adhesive tape residues. The
30 compositions of the invention may also be used to test the
quality of curing of ultraviolet soldermask.

35

-2-

BACKGROUND OF THE INVENTION

5 In the fabrication of printed wiring boards and/or printed circuit boards, soldering fluxes are first applied to the substrate board material to ensure firm, uniform bonding of the solder. These soldering fluxes fall into two broad categories: rosin and non-rosin, or water soluble, fluxes. The rosin fluxes, which are generally non-corrosive and have a much longer history of use, are still widely used throughout the electronics industry. The water soluble
10 fluxes, which are a more recent development, are being used increasingly in consumer products applications. Because they contain strong acids and/or amine hydrohalides and are thus corrosive, the water soluble soldering fluxes can cause circuit failure if residual traces of the material are not
15 carefully removed. For that reason military specifications require the use of rosin fluxes.

Even the use of rosin soldering fluxes, however, can lead to premature circuit failure due to decreased board
20 resistance if traces of residual flux are not removed following soldering. While water soluble fluxes can be easily removed with warm, soapy water, the removal of rosin flux from printed wiring boards has traditionally been carried out with the use of chlorinated hydrocarbon solvents
25 such as 1,1,1,-trichlorethane, trichloromonofluoromethane, methylene chloride, trichlorotrifluoroethane, or mixtures or azeotropes of these solvents. These solvents are undesirable, however, because they are toxic. Thus, their use is subject to close scrutiny by the Occupational Safety
30 and Health Administration (OSHA), and stringent containment equipment must be used. Moreover, if released into the environment these solvents are not readily biodegradable and are thus hazardous for long periods of time.

35

-3-

Alkaline cleaning compounds known as the alkanol amines, usually in the form of monoethanolamine, have been used for rosin flux removal as an alternative to the toxic chlorinated hydrocarbon solvents. These compounds chemically react with rosin flux to form a rosin soap through the process of saponification. Other organic substances such as surfactants or alcohol derivatives may be added to these alkaline cleaning compounds to facilitate the removal of such rosin soap. Unfortunately, these compounds like the water soluble soldering fluxes have a tendency to cause corrosion on the surfaces and interfaces of printed wiring boards if they are not completely and rapidly removed during the fabrication process.

In another approach, Bakos et al. [U.S. Patent No. 4,276,186] have used mixtures of N-methyl-2-pyrrolidone and a water miscible alkanolamine to remove solder flux and solder flux residue from integrated circuit modules. These mixtures were also said to be useful for removing various cured synthetic organic polymer compositions such as cured polyimide coating compositions from integrated circuit chip modules.

During the manufacture of printed wiring boards, it is sometimes necessary to temporarily protect certain portions of the board from processing steps such as the process of creating corrosion resistant gold connecting tabs at the board edges. This transient protection of portions of the circuit board can be achieved by the application of special adhesive tape to susceptible areas. Once such protection is no longer needed, the adhesive tape must be removed. A residue of the tape adhesive generally remains which, if not thoroughly removed, can cause premature board failure. Removal of this tape residue has traditionally been

35

-4-

carried out by the use of chlorinated solvents which, as already described, are toxic and environmentally undesirable.

5 A further procedure that is commonly carried out during printed circuit board fabrication is the application of a soldermask. As the name implies, a soldermask is a polymer or resin coating that is selectively applied to a printed circuit board to shield areas where solder is not required or desired. Where such masking is to be permanent, the soldermask must be "cured," a process by which monomeric
10 reactants are made to polymerize. If curing of the soldermask is incomplete, solder will penetrate areas that are to be shielded, resulting in destruction of the board.

15 One kind of soldermask is based on acrylate chemistry and is photo-initiated and polymerized, and thus cured, using ultraviolet (U.V.) light. The use of U.V.-cured soldermask is advantageous in that its curing is rapid, but the thickness of such soldermask films must be uniform for successful curing. The U.V. irradiation used cannot
20 penetrate to the bottom of deep soldermask deposits, and attempts to increase the intensity or the duration of irradiation to cure thick sections can result in the embrittlement of the thinner mask film regions.

25 SUMMARY OF THE INVENTION

It is an object of the present invention to provide compositions and methods for the safe and effective removal of rosin soldering fluxes from printed wiring boards without
30 otherwise adversely affecting the boards. It is a further objective of this invention to provide safe and effective compositions and methods for the removal of adhesive tape residues from printed wiring boards. It is a still further objective of the present invention to provide methods and
35

-5-

compositions to detect improperly cured U.V. soldermask and to thus avoid printed wiring board failure on that account.

5 This invention provides cleaning methods and compositions for the removal of rosin solder fluxes and adhesive tape residues during the fabrication of printed circuit or wiring boards. As a result, the possibility of premature circuit failure that might occur in the absence of such cleaning is eliminated or greatly reduced. The cleaning efficacy of the compositions of the invention is such that
10 printed wiring boards thus treated meet stringent U.S. Department of Defense specifications.

The compositions of the invention can also be used to detect improperly cured U.V. soldermask. This task is
15 accomplished by exploiting an ability of the compositions to selectively lift inadequately cured regions of such soldermask from the board, while leaving properly cured regions intact.

20 The compositions of the invention are characterized by low toxicity, biodegradability and non-corrosiveness, unlike the chlorinated hydrocarbon solvents and alkaline cleaners that have heretofore been employed for printed wiring board and printed circuit board cleaning. As a result, the need
25 for costly containment equipment is eliminated.

More particularly, the present invention provides printed circuit/wiring board treatment compositions comprising terpene compounds such as limonene or dipentene.
30 These terpene compounds, which have the ability to dissolve or complex with and remove rosin solder fluxes, oils, waxes and greasy substances or adhesive tape residues, could be used as such and removed, e.g., with clean absorbent materials. Preferably, however, the terpene compounds of the
35

-6-

invention are combined with terpene emulsifying surfactants to facilitate removal by water.

BRIEF DESCRIPTION OF THE FIGURE

5 The present invention may be more readily understood by reference to FIG. 1, wherein a flow chart shows a representative printed circuit/wiring board fabrication process.

10 DETAILED DESCRIPTION OF THE INVENTION

 The compositions of the present invention contain terpene compounds that have the capability to dissolve rosin soldering flux or adhesive tape residues that are commonly
15 used in the fabrication of printed circuit or wiring boards. Terpene compounds that are suitable for this purpose include but are not limited to pinene, both alpha and beta isomers, gamma terpinene, delta-3-carene, limonene and dipentene (the racemic mixture of the isomers of optically active limonene),
20 with limonene and dipentene being preferred.

 These terpene compounds could be used alone and removed, after allowing them to complex with and dissolve rosin flux or adhesive tape residues, e.g., by flushing with
25 excess terpenes or by absorption into paper or cloth. Because they are almost completely insoluble in water, however, the terpenes cannot be directly flushed away by water. Alternatively and preferably, the terpene compounds are combined with one or more terpene emulsifying
30 surfactants. The addition of such surfactants facilitates removal of the terpenes from printed wiring boards by rinsing with water, whereby the terpenes are formed into oil-in-water emulsions.

35

-7-

Numerous surfactants capable of emulsifying the terpenes of the invention may be used, including but not limited to the linear alkyl benzene sulfonates, linear or branched chain alcoholic ethoxylates and ethoxysulfates, polysorbate esters, ethoxylated alkylphenols and alkyl and dialkyl succinate compounds. An example of the latter class of compounds is sodium dioctyl sulfosuccinate. The ethoxylated alkylphenols contain various alkyl side chains and various numbers of linked ethylene oxide units. Useful compounds of this class contain from about 5 to about 20 ethylene oxide groups, with 7 or 8 being preferred.

The quantity of terpene emulsifying surfactants in the compositions of the present invention may range on a weight basis from 0 to about 40%, with the terpene compounds accounting for the balance of the compositions. In a preferred embodiment designated EC-1, the composition comprises by weight about 6.5% poly (7) ethoxy nonylphenol, 2.1% poly (10) ethoxy nonylphenol, 1.4% sodium dioctyl sulfosuccinate and 90% limonene.

The compositions of this invention are characterized by low toxicity and environmental biodegradability, unlike the chlorinated hydrocarbon solvents that had been used prior to this invention for printed wiring board cleaning. Limonene, for example, is a natural product in various ethereal oils such as oils of lemon, orange, caraway, dill and bergamot which possesses a pleasant, lemon-like odor. EC-1 has biological oxygen demand and chemical oxygen demand values of 295 and 1,425 milligrams per liter, respectively, at a dilution of 1,000:1, as determined by methods described in Standard Methods for the Examination of Water and Wastewater, 16th Edition, American Public Health Association.

35

-8-

The applicability of the compositions of the invention to various aspects of the printed circuit/wiring board fabrication process can best be understood by reference to a representative process flow chart, which is shown in Figure 1.

5 The assembly manufacturing process involves the placement of components such as integrated circuits, resistors, capacitors, diodes, etc. on the surface of the board or their insertion through pre-drilled holes. The components are then secured by soldering by mechanical or
10 automatic means. Interspersed with the soldering operations are cleaning procedures and inspections to ensure that tape and solder flux residues that could lead to premature circuit failure do not remain. Points in the fabrication process to which the compositions of the invention apply will be
15 identified below.

ROSIN FLUX CLEANING AND ADHESIVE TAPE RESIDUE
REMOVAL

20 For the removal of rosin soldering flux deposits or adhesive tape residues during printed circuit/wiring board fabrication, the compositions of the invention may be applied to the boards by immersion in dip tanks or by hand or mechanical brushing. Alternatively, they may be applied by
25 any of the commercially available printed wiring board cleaning equipment. Dishwasher size units may be employed, or much larger continuous belt cleaning machinery such as Total Systems Concept's Dual Process Cleaning System models 31-218, 31-418, 31-224 and 31-424 (Total Systems Concept, St.
30 Louis, MO). Depending upon their design, these washers may apply the compositions of the invention by spraying with mechanical nozzles or by rolling contact with wetted roller surfaces. The temperature at which the compositions may be applied can range from room, or ambient, temperature (about
35

-9-

70°F) to about 150°F. The compositions containing terpene emulsifying surfactants may be used full strength or diluted with water to as low as about a 2 volume percent concentration. The compositions should contact the boards for about 1 to about 5 minutes.

5

The points at which the compositions of the invention would be applied for solder flux removal in a typical printed wiring board fabrication process are indicated in Fig. 1 by the term "post clean," referring to the post solder cleaning process.

10

Once solder flux has been loosened and solubilized during a period of contact which typically ranges from about 1 to about 5 minutes, the compositions of the invention are removed. Where the terpene compounds are used alone, their removal can be carried out by flushing with a non-toxic, miscible solvent or by absorption into appropriate materials. In the preferred embodiments of the invention, which contain terpene emulsifying surfactants, the boards may simply be flushed with water for a period of up to about 2 minutes. Deionized water is preferred. The optimal rinsing time varies according to the kinds of surfactants and the concentrations of the terpene formulations used and can easily be determined by routine experimentation.

15

20

25

The cleaned boards are then dried, preferably with forced air. Drying is expedited if the air is warmed, preferably to above about 100°F.

30

The efficacy of rosin soldering flux removal from printed wiring boards is such that the boards meet stringent military specifications for low resistivity after cleaning. For example, the boards meet the Mil-P-28809A standard for low resistivity of solvent extracts by which contamination

35

-10-

has been removed from a cleaned board according to Mil-P-55110C. The resistivity of solvent extracts of cleaned boards is most easily determined with an Omega Meter. Omega Meter is the registered trademark of Kenco Industries, Inc., Atlanta, GA, for a microprocessor-controlled contamination test system that rapidly measures changes in resistivity due to contaminating ions.

The result of Omega Meter measurements are expressed in equivalent units of mg NaCl/in² or its metric equivalent. According to MIL-P-28809A, the acceptable resistivity value for a cleaned board is equivalent to 2.2 ug NaCl/cm² or 14 ug NaCl/in², but far better results are routinely obtained after solder flux has been removed with the compositions of the present invention. A value of about 0.67 ug NaCl/cm² or 4.3 ug NaCl/in² is typical.

During the process of gold plating connecting tabs to improve corrosion resistance, tin-lead residues must first be removed from the unplated tabs. Removal of these residues is carried out through the use of etching chemicals that can damage other unprotected printed circuit/wiring board components. To protect vulnerable components from the etching chemicals, boards are wrapped on both sides with an adhesive plating tape which forms a shield or splash guard for all but the exposed tab area. The etching chemicals then remove the tin-lead residues on the tabs, a nickel plate is applied as a base for the gold, and gold plating of the tabs is finally carried out. The adhesive plating tape which was maintained in place through all of these etching and plating steps, is then removed. The use of adhesive tape is indicated in Fig. 1 at the point labeled "tape for gold plating." The tape is removed following the "nickel and gold plate" step, and it is at this point that the terpene

-11-

compositions of the invention may most advantageously be used.

5 Following removal of the tape, a silicone-based and/or rubber-based residue may remain on the board. This residue may easily be removed by the compositions of the invention under the same conditions described above for solder flux removal. The exact operational parameters will be determined by the nature of the tape residue and the tenacity with which it adheres to the board, but the
10 conditions described above are generally effective. As in the case of solder flux removal, treatment of the board with the terpene compounds of the invention is generally followed by water flushing and air drying.

15 The efficiency of removal of adhesive tape residues from printed circuit/wiring boards by the compositions of the invention is such that no residues are visible after cleaning. A simple 5-10X stereomicroscope can facilitate visual inspection for tape residues following cleaning.

20

QUALITY CONTROL TESTING FOR IMPROPERLY CURED
U.V. SOLDERMASK

25 It has unexpectedly been found that the compositions of the invention will cause a lifting or bubbling of uncured areas U.V. curable soldermask on printed circuit/wiring boards, while leaving properly cured regions undisturbed. The detection of improperly cured soldermask in this fashion can be beneficial by eliminating the use of such
30 defective boards before valuable components are placed thereon. Such detection is carried out at the "Board Compete and Ready for Assembly" point in Fig. 1.

35

-12-

Soldermask to which such detection is applicable is based upon methacrylate polymer chemistry. Such polymers, upon exposure to ultraviolet light, become "cured" through the photoinitiated cross-linking of the polymers to produce a tough protective coating that resists subsequent solder adherence. It is poorly crosslinked regions of the soldermask that are susceptible to disruption by the terpene compounds of the invention.

The detection of such uncured soldermask regions can be carried out by suspending printed circuit/wiring boards to which a U.V. soldermask has been affixed and cured in a composition such as EC-1 for a period of time, rinsing in water and then making a visual inspection. Submersion in the cleaning solution or spray application etc. for from about 1 to about 5 minutes at a temperature of from about 70 to about 150°F is usually sufficient. The boards are then generally flushed with water and dried as described above for solder flux and adhesive tape residue removal. Where dip tanks are used, a second tank that is continually flushed with fresh water is preferably used to rinse the boards.

Inspection can be facilitated by the use of a microscope such as a 5-10X stereomicroscope, but the use of such an instrument is not essential. Boards having improperly cured U.V. solder mask show evidence of blisters, bubbles, discoloration, cracks and deformation in the defective regions.

EXAMPLE: ROSIN FLUX CLEANING

To illustrate the cleaning ability of the compositions of the invention, printed wiring boards that had been wave soldered using rosin flux conforming to MIL-F-14256 (type RMA) were cleaned in a mechanical cleaning system.

-13-

The cleaning composition, designated EC-1, contained by weight 90% d-limonene, 6.5% poly (7) ethoxy nonylphenol, 2.1% poly (10) ethoxy nonylphenol and 1.4% sodium dioctyl sulfosuccinate. Concentrations of 25 to 100 volume percent of EC-1 diluted in water were used as indicated below. The cleaning system was a Series 400, Model TRL-HSE Aqueous Cleaning System manufactured by The John Treiber Company, Huntington Beach, CA.

The cleaning system has a conveyor driven modular design in which the operations of loading, pre-wash rinsing, washing, air knife drying, first rinsing, air knife drying, final rinsing and high speed drying are carried out in succession. Rinse water at ambient temperature and EC-1 for washing at 120°F were applied through spray nozzle manifolds as the boards traversed the system at a conveyor speed of 8 feet/minute. In air knife drying, turbine propelled air shears fluids from the board surfaces. In the final high speed drying module, two sets of dual air knives above and below the conveyor thoroughly dry the cleaned boards. The air in this module was heated to 120°F.

Cleaned and dried boards were evaluated for cleaning efficiency by Omega Meter resistivity measurements. To make these measurements, cleaned and dried boards were loaded into a test cell of the instrument and then extracted with a circulating solution of isopropanol: water (25:75, v/v) as specified by MIL-P-55110C and MIL-P-28809A. The resistivity of the solution was measured at a rate of 24 times per minute over a period of about 5-15 minutes until equilibrium was reached, indicating that extraction of board surface contamination was essentially complete. Equilibrium was defined as the point at which the change in measured resistivity of the solution was less than or equal to 5% of any value measured in the previous two minutes.

-14-

The equilibrium resistivity measurements for a number of cleaning tests are shown in Table 1.

TABLE 1

RESISTIVITY MEASUREMENTS OF EC-1 CLEANED
ROSIN FLUX SOLDERED PRINTED WIRING BOARDS

Test Number	EC-1 Concentration (volume %)	Equivalent NaCl Contamination (ug/in ²)
1	25	4.7
2	25	4.1
3	50	4.1
4	50	4.7
5	75	3.9
6	75	4.0
7	100	4.6

As shown in Table 1, EC-1 at all concentrations examined were effective in producing levels of residual board surface contamination that were far below the MIL-P-28809A requirement of 14 ug NaCl/in² equivalent.

Many modifications and variations of this invention may be made without departing from its spirit and scope, as will become apparent to those skilled in the art. The specific embodiments described herein are offered by way of example only, and the invention is limited only by the terms of the appended claims.

WHAT IS CLAIMED IS:

1. A method for removing rosin soldering flux or adhesive tape residues from a printed wiring board, comprising
- (a) contacting the board with a composition comprising a terpene compound and from 0 to about 40% by weight of a terpene emulsifying surfactant;
- (b) allowing the contact to continue for sufficient time to solubilize the soldering flux or tape residues; and
- (c) removing the composition and solubilized soldering flux or tape residues from the board.
2. The method of claim 1 wherein the terpene emulsifying surfactant is selected from the group consisting of linear alkyl benzene sulfonates, linear or branched chain alcoholic ethoxylates and ethoxysulfates, polysorbate esters, alkyl and dialkyl succinate compounds, and ethoxylated alkylphenols, which ethoxylated alkylphenols have from about 5 to about 20 ethylene oxide groups.
3. The method of claim 1 wherein the terpene compound is selected from the group consisting of limonene and dipentene.
4. The method of claim 2 wherein the terpene compound is selected from the group consisting of limonene and dipentene.
5. The method of claim 3 wherein the terpene compound is limonene.

-16-

6. The method of claim 3 wherein the terpene compound is dipentene.

5 7. The method of claim 5 wherein the surfactant comprises by weight of the composition about 6.5% poly (7) ethoxy nonylphenol, 2.1% poly (10) ethoxy nonylphenol and 1.4% sodium dioctyl sulfosuccinate.

10 8. The method of claim 1 wherein the contact is carried out at a temperature of from about room temperature to about 150°F.

15 9. The method of claim 2 wherein the contact is carried out at a temperature of from about room temperature to about 150°F.

10. A method for detecting improperly cured U.V. soldermask on a printed wiring board, comprising

- 20 (a) contacting the board with a composition comprising a terpene compound and from 0 to about 40% by weight of a terpene emulsifying surfactant;
- (b) allowing the contact to continue for sufficient time to visibly loosen improperly cured U.V. soldermask areas;
- 25 (c) removing the composition from the board; and
- (d) detecting the visibly loosened improperly cured soldermask areas.

30 11. The method of claim 10 wherein the terpene emulsifying surfactant is selected from the group consisting of linear alkyl benzene sulfonates, linear or branched chain alcoholic ethoxylates and ethoxysulfates, polysorbate esters, alkyl and dialkyl succinate compounds, and ethoxylated

35

-17-

alkylphenols, which ethoxylated alkylphenols have from about 5 to about 20 ethylene oxide groups.

5 12. The method of claim 10 wherein the terpene compound is selected from the group consisting of limonene and dipentene.

10 13. The method of claim 11 wherein the terpene compound is selected from the group consisting of limonene and dipentene.

 14. The method of claim 12 wherein the terpene compound is limonene.

15 15. The method of claim 12 wherein the terpene compound is dipentene.

20 16. The method of claim 14 wherein the surfactant comprises by weight of the composition about 6.5% poly (7) ethoxy nonylphenol, 2.1% poly (10) ethoxy nonylphenol and 1.4% sodium dioctyl sulfosuccinate.

25 17. The method of claim 10 wherein the contact is carried out at a temperature of from about room temperature to about 150°F.

 18. The method of claim 11 wherein the contact is carried out at a temperature of from about room temperature to about 150°F.

30

35

AMENDED CLAIMS

[received by the International Bureau on 7 October 1986 (07.10.86)
original claims 3-6,12-15 amended; remaining claims unchanged
(4 pages)]

- 5 1. A method for removing rosin soldering flux
or adhesive tape residues from a printed wiring board,
comprising
- 10 (a) contacting the board with a composition
comprising a terpene compound and from 0 to
about 40% by weight of a terpene
emulsifying surfactant;
- 15 (b) allowing the contact to continue for
sufficient time to solubilize the soldering
flux or tape residues; and
- (c) removing the composition and solubilized
soldering flux or tape residues from the
board.
- 20 2. The method of claim 1 wherein the terpene
emulsifying surfactant is selected from the group
consisting of linear alkyl benzene sulfonates, linear or
branched chain alcoholic ethoxylates and ethoxysulfates,
polysorbate esters, alkyl and dialkyl succinate compounds,
and ethoxylated alkylphenols, which ethoxylated
25 alkylphenols have from about 5 to about 20 ethylene oxide
groups.
- 30 3. The method of claim 1 wherein the terpene
compound is selected from the group consisting of the
d-form of limonene, the l-form of limonene and dipentene.
- 35 4. The method of claim 2 wherein the terpene
compound is selected from the group consisting of the
d-form of limonene, the l-form of limonene and dipentene.

5. The method of claim 1 wherein the terpene compound is the d-form of limonene or the l-form of limonene.

5 6. The method of claim 1 wherein the terpene compound is dipentene.

7. The method of claim 5 wherein the surfactant comprises by weight of the composition about 6.5% poly (7) ethoxy nonylphenol, 2.1% poly (10) ethoxy nonylphenol and 1.4% sodium dioctyl sulfosuccinate.

8. The method of claim 1 wherein the contact is carried out at a temperature of from about room temperature to about 150°F.

9. The method of claim 2 wherein the contact is carried out at a temperature of from about room temperature to about 150° F.

20

10. A method for detecting improperly cured U.V. soldermask on a printed wiring board, comprising

- (a) contacting the board with a composition comprising a terpene compound and from 0 to about 40% by weight of a terpene emulsifying surfactant;
- (b) allowing the contact to continue for sufficient time to visibly loosen improperly cured U.V. soldermask areas;
- (c) removing the composition from the board; and
- (d) detecting the visibly loosened improperly cured soldermask areas.

25

30

35

11. The method of claim 10 wherein the terpene emulsifying surfactant is selected from the group consisting of linear alkyl benzene sulfonates, linear or branched chain alcoholic ethoxylates and ethoxysulfates, polysorbate esters, alkyl and dialkyl succinate compounds, and ethoxylated alkylphenols, which ethoxylated alkylphenols have from about 5 to about 20 ethylene oxide groups.

12. The method of claim 10 wherein the terpene compound is selected from the group consisting of the d-form of limonene, the l-form of limonene and dipentene.

13. The method of claim 11 wherein the terpene compound is selected from the group consisting of the d-form of limonene, the l-form of limonene and dipentene.

14. The method of claim 10 wherein the terpene compound is the d-form of limonene or the l-form of limonene.

15. The method of claim 10 wherein the terpene compound is dipentene.

16. The method of claim 14 wherein the surfactant comprises by weight of the composition about 6.5% poly (7) ethoxy nonylphenol, 2.1% poly (10) ethoxy nonylphenol and 1.4% sodium dioctyl sulfosuccinate.

17. The method of claim 10 wherein the contact is carried out at a temperature of from about room temperature to about 150°F.

18. The method of claim 11 wherein the contact is carried out at a temperature of from about room temperature to about 150°F.

5

10

15

20

25

30

35

STATEMENT UNDER ARTICLE 19

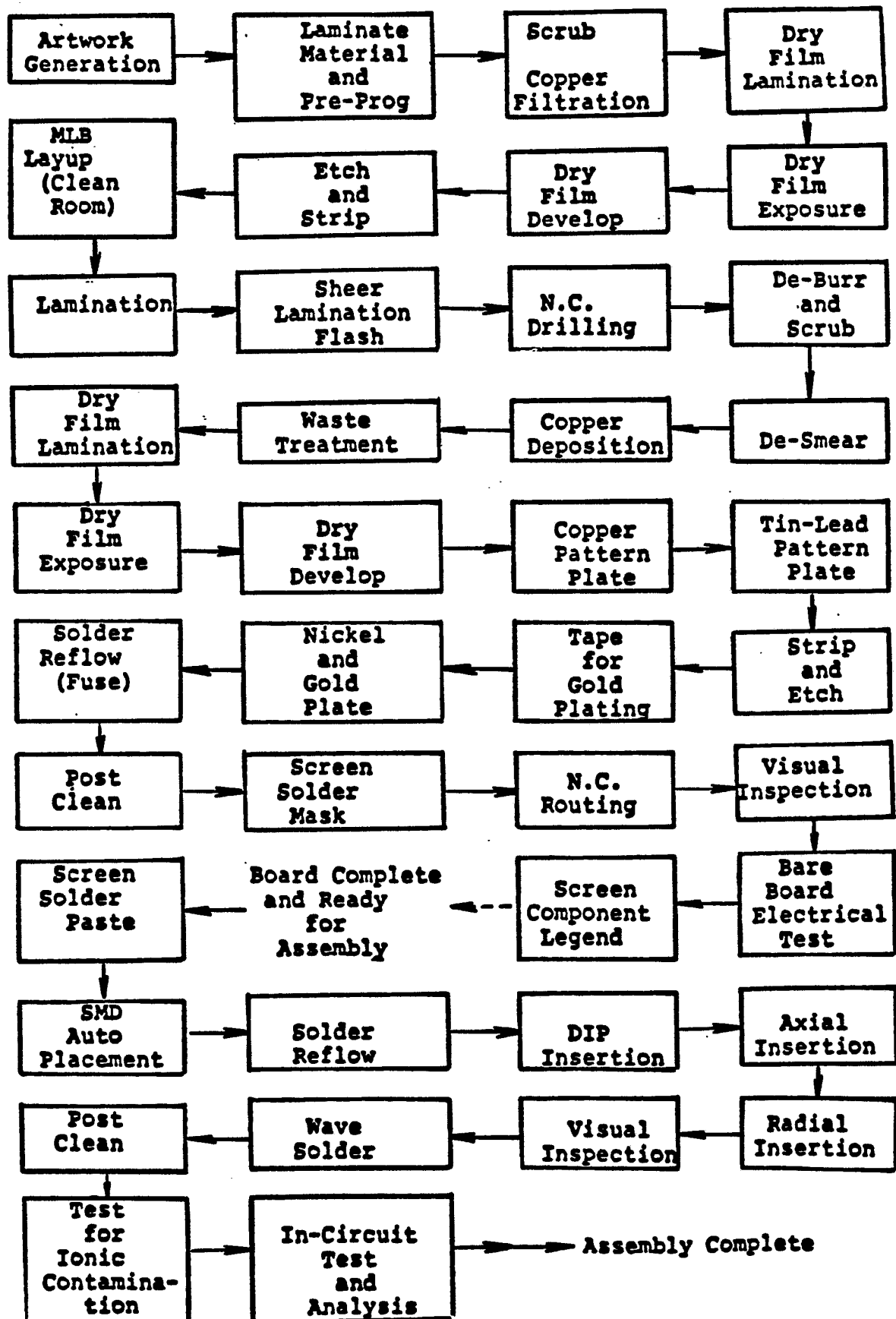
The reasons for the foregoing amendments are as set forth below:

The dependencies of claims 5, 6, 14 and 15 were changed to correct editorial oversights.

The change to claims 3, 4, 5, 12, 13 and 14, i.e., the substitution of terminology specifying the d-form and l-form of limonene for the single word "limohene", was made to conform the claims of the International Application with the allowed claims of the corresponding U.S. Application Serial No. 750,198 filed July 1, 1985. The amendment to the corresponding claims of the U.S. application was made at the request of the U.S. Examiner, indicating he did not consider said amendment to go beyond the disclosure of the corresponding U.S. application as filed. In view of this fact and in view of the disclosure at page 6, lines 19-20, it is submitted that the foregoing amendments to the claims of the International Application do not run afoul of PCT Article 19, subsection (2).

1/1

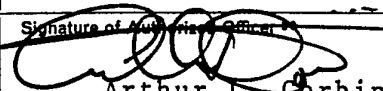
FIGURE 1



SUBSTITUTE SHEET

INTERNATIONAL SEARCH REPORT

International Application No **PCT/US86/01361**

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ³		
According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC (4): C23G 1/00		
U.S. CL. 134/40, 42		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁴		
Classification System	Classification Symbols	
U.S.	134/26, 40, 42	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁵		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ¹⁴		
Category *	Citation of Document, ¹⁶ with indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No. ¹⁸
Y	US, A, 4,246,147 (BAKOS et al) 20 January 1981, See col. 1, lines 7-15; col. 2, lines 49-54; col. 4, lines 1-20 and col. 8, lines 1-28.	1-18
Y	US, A, 4,276,186 (BAKOS et al) 30 June 1981, See col. 1, lines 5-15; col. 4, lines 17-21 and 61-67 and col. 5, lines 5-13.	1-18
Y	US, A, 4,294,729 (BAKOS et al) 13 October 1981, See cols. 2, 3, 6 and 7.	1-18
A	US, A, 4,369,287 (HUTCHINSON et al) 18 January 1983, See entire document.	1-18
A	US, A, 4,373,974 (BARAJAS) 15 February 1983, See entire document.	1-18
Y	US, A, 4,434,084 (HICKS et al) 28 February 1984, See col. 4, lines 1-12.	1-18
Y	US, A, 4,511,488 (MATTA) 16 April 1985, See cols. 2 and 3.	1-18
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>* Special categories of cited documents: ¹⁵</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"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)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"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</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"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.</p> <p>"&" document member of the same patent family</p> </div> </div>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search ²		Date of Mailing of this International Search Report ³
08 September 1986		11 SEP 1986
International Searching Authority ¹		Signature of Authorizing Officer ¹
ISA/US		 Arthur L. Corbin