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LOW-RESIDUE SOLDERING FLUX CONTAINING ROSIN

This invention relates to a low-residue soldering flux, especially a flux containing a dibasic acid mixture, that, in use, is essentially non-corrosive and ionic residue-free.

5 A soldering flux is used to aid fusion of metal parts being joined together by soldering, by reacting with and dissolving the surface oxides or impurities thereon whilst coating the surfaces against oxidation, during soldering at a temperature
10 above the solder melting point.

 Organic fluxes are typically based on water-insoluble rosin or a water-soluble organic acid. Activated rosin fluxes are used in soldering electrical connections on printed circuit boards.
15 Wave-soldering is used for mass production circuit board soldering as by applying the flux, pre-heating the board, applying the solder (e.g., via a molten solder standing wave), cooling the board, and cleaning it to remove flux residue therefrom.

20 Such flux residue is mainly constituted of ionic (e.g., acidic or basic) substances, and is corrosive, or can hydrolyze to corrosive constituents in the presence of moisture (e.g., air moisture). This can lead to short-circuits, noise generation,
25 etc., in use of the circuit board product. These adverse results are effectively avoided by subjecting the soldered board to a cleaning step to remove the ionic substances therefrom.

 U.S. Patent No. 2,715,084 (Konig) shows a
30 flux of rosin activated by a chain-brominated higher aliphatic monocarboxylic acid or salt of 8-24 chain carbon atoms (e.g., alpha-bromolauric acid), in turpentine, that does not decompose at the soldering

temperature. It is non-corrosive as the chain bromine is not ionic so that the compound does not hydrolyze to a corrosive halide, as with acid or halide salt flux, or rosin flux activated by organic base halide (e.g., cetyl-pyridinium halide), wherein the halide as an anion can hydrolyze in air to form a corrosive acid halide. Brominated acids of less than 8 carbon atoms are excluded as being corrosive and of less surface activity.

10 U.S. Patent No. 2,898,255 (Thompson et al.) shows a non-corrosive printed circuit board flux of rosin activated by a combination of both an aliphatic monocarboxylic acid (e.g., formic, acetic or propionic acid) and an aliphatic dicarboxylic acid
15 (e.g., oxalic, malonic, succinic, glutaric or adipic acid), in isopropanol, that does not splatter during soldering. The sole example given of a non-splattering flux is a mixture of rosin (mp 120°C/248°F), formic acid (bp 100°C/212.5°F) and
20 glutaric acid (bp 200°C/392°F), in isopropyl alcohol, in which the low glutaric acid content (about 1%) is said to prevent splattering, whilst the given melting and boiling points of the components are said to permit step-by-step de-oxidation of the oxide surface
25 being soldered.

U.S. Patent No. 2,904,459 (Kubota) shows a liquid, paste or solid flux for electrical application. The flux is based on rosin converted to pure, stable abietic acid amine salt (e.g.,
30 diethylene amine salt), that is insoluble and does not hydrolyze, and whose neutral resinous residue on soldering is non-corrosive and non-hygroscopic, unlike commercial rosin which, besides a 25-30%

content of abietic acid (i.e., a monobasic organic acid), that has fluxing action, also has a resinous remainder that can cause soldered product defects.

5 U.S. Patent Nos. 3,235,414 (Marks) and 3,264,146 (Marks) show a rosin-free food can soldering flux. It is formed as a relatively concentrated solution of a large percentage (e.g., 25 wt. parts) of an aliphatic dicarboxylic acid of 9-10 carbon atoms with at least 4 carbon atoms in the
10 chain separating the carboxyl groups (e.g., azelaic or sebacic acid), or its anhydride or ester, that is modified by (e.g., 1.2 wt. parts) ethanolamine hydrochloride, in (e.g., 75 wt. parts) dimethylformamide as polar solvent. Such acids with
15 3 or less carbon atoms separating the carboxyl groups present in the molecule are undesired in forming monoanhydrides from a single molecule.

U.S. Patent No. 3,837,932 (Aronberg) shows a 10-80% polycarboxylic acid particulate suspension
20 (e.g., adipic acid), in an aqueous solution of 0.2-5% soluble gum (remainder 15-89.8% water), as a rosin-free soldering flux for bonding lead storage battery plate lugs to straps, in which the acid (e.g., adipic) does not sublime at the high
25 (482°C/900°F) bonding temperature used. Use of the dry particulate acid alone, applied to the lugs after pre-heating, is less preferred.

U.S. Patent No. 4,168,996 (Zado) shows a mixture of rosin, an activator with both a halogen
30 and a destabilizing substituent, such as a

halogenated mono or dibasic organic acid (e.g., 2,3-dibromosuccinic acid), and a solder surfactant such as a polycarboxylic acid (e.g., adipic, sebacic or azelaic acid), or hydroxyl substituted polybasic acid, and/or keto acid (e.g., levulinic acid), in ethanol or isopropyl alcohol, and optionally a secondary activator (e.g., diethylamine hydrochloride) and a non-ionic foaming agent, as being activated rosin flux of synergistic effect for the soldering of printed circuit boards.

Zado notes that using rosin with the activator or solder surfactant alone does not provide the synergistic effect, and that the combination acid containing rosin flux of the above-discussed U.S. patent 2,898,255 is too acidic and corrosive for practical use in electronics soldering.

The importance of a residue-free soldered connection is discussed in the article entitled: "Europeans find ways to phase-out CFCs," Electronic Packaging & Production, Vol. 29, No. 1, Jan. 1989, pp. 26 and 28, Linda Smith-Vargo, Associate Editor. The article notes that fluxes used in printed circuit board applications leave residues that must be cleaned, typically by using an organic liquid based on fully halogenated chlorofluorocarbons or CFCs, but that in view of the need to reduce CFC emissions, due to environmental constraints, soldering procedures are needed that avoid CFCs yet produce a residue-free non-corrosive product.

It is desirable to have a flux for soldering metal connections in electrical

applications such as printed circuit boards that does not produce a corrosion-causing and/or hydrolyzable ionic residue that requires a cleaning step for its removal.

5 This invention solves the foregoing problems by providing a low-residue soldering flux and, particularly, a flux containing a dibasic acid mixture or three-acid mixture (TAM) that in use is essentially non-corrosive and ionic residue-free.

10 All percentages (%), parts (pts) or proportions set forth herein are by weight unless specifically indicated otherwise.

 The low-residue flux of the invention is used in heat-soldering metal components, and the
15 three-acid mixture is an essentially non-subliming dibasic acid mixture of succinic, glutaric and adipic acids. It contains 15-30% succinic acid, 40-63% glutaric acid and 10-30% adipic acid and, preferably, 18-30% succinic acid, 51-61% glutaric acid and 11-30%
20 adipic acid. The percentages of succinic, glutaric and adipic acid are based on the total dibasic acid mixture.

 The three-acid mixture desirably has a melting point (mp) of 90-130°C and a boiling point
25 (bp) of 150-210°C, especially 170-195°C, and is capable of essentially completely volatilizing during heat-soldering to leave the soldered site essentially free from ionic residue. In particular, it is capable of leaving at most a trace amount of a residue of
30 corrosive, or hydrolyzable potentially corrosive, ionic (e.g., acidic) constituents.

 The soldered site such as an electronic circuit board needs no cleaning to remove the ionic residue (e.g. by chlorofluorocarbons or like

environmentally-objectionable washing liquids), and may be further processed without a cleaning step.

As used herein, essentially "non-subliming" means that the constituents of the three-acid mixture in the flux that volatilize during the heat-soldering operation, remain volatilized and do not re-condense and/or re-deposit at the soldering site as ionic residue. Thus, the three-acid mixture is an essentially non-re-condensing and non-re-depositing volatilizable dibasic acid mixture according to the invention.

The flux is a rosin flux comprising rosin and an effective activating amount of said three-acid mixture.

As to the rosin flux, the rosin and three-acid mixture may be in the relative proportion to each other of 65-95% rosin and 5-35% three-acid mixture (i.e., in the ratio of 65-95 parts rosin to 35-5 parts three-acid mixture). The flux may include a volatilizable flux vehicle, such as with the rosin and three-acid mixture being in a combined amount of 1.25-55% and the vehicle in an amount of 45-98.75%. The vehicle is also capable of essentially completely volatilizing to leave the site essentially ionic residue-free, and may be an organic solvent such as an aliphatic or cycloaliphatic alcohol, aliphatic carboxylic acid ester, cyclic ketone, or mixture thereof. Soldering of metal surfaces with such a flux is carried out at a temperature sufficient to leave at most a trace amount of ionic residue on said metal surfaces.

The vehicle of the rosin flux may optionally contain a minor diluent amount of de-ionized water, and thus may comprise 70-100% solvent and 0-30% de-ionized water. The solvent and water may be

typically in the relative proportion to each other of 70-99.5% solvent and 0.5-30% water (i.e., in the ratio of 70-99.5 parts solvent to 30-0.5 parts water). As used herein, a "minor" amount of water means at most
5 about 30% of the total vehicle.

Typically, the rosin flux of the present invention consists essentially of 1-35% rosin, 0.25-20% of said three-acid mixture, 45-98.75% volatilizable organic solvent and 0-20% de-ionized
10 water. The percentages of rosin, three-acid mixture, solvent and water are based on the total flux.

In a high rosin content flux, such as for general metal soldering, the flux of the invention typically contains 25-35% rosin, 2.5-20% three-acid
15 mixture, 45-60% solvent and 5-20% water. The solvent and water are typically in the relative proportion to each other of 75-90% solvent and 10-25% water (i.e., in the ratio of 75-90 parts solvent to 25-10 parts water). A method of heat-soldering metal surfaces

using such a flux comprises applying a solder to the metal surfaces in the presence of an effective amount of said low-residue flux in coating disposition on the metal surfaces, and at a temperature sufficient for molten flow of the solder and for volatilization of the flux from the metal surfaces, so as to leave the metal surfaces essentially free from ionic residue from the flux. In a low rosin content flux, such as for circuit board component soldering, the flux of the invention typically contains 1-8% rosin, 0.25-1% three-acid mixture, 90-98.75% solvent and 0-1% water. The solvent and water are typically in the relative proportion to each other of 99-99.5% solvent and 0.5-1% water (i.e., in the ratio of 99-99.5 parts solvent to 1-0.5 parts water).

The organic solvent may be an alcohol such as a lower boiling or lower aliphatic alcohol, typically a lower alkyl alcohol or lower alkanol, such as ethyl, n-propyl, isopropyl, n-butyl, or sec-butyl, alcohol, or higher boiling aliphatic or cycloaliphatic alcohol, such as tetrahydrofurfuryl alcohol (about bp 178°C), and the like, or other such volatilizable aliphatic or cycloaliphatic alcohols in which the flux contents are soluble. As used herein, "lower" aliphatic, alkyl or alkanol means a chain of less than 5 carbon atoms. The alcohol is typically isopropyl alcohol (sp.g. 0.78), and it may be added to the rosin flux along with the rosin, as where a commercial rosin solution there is used.

The organic solvent may also be any other

appropriate solvent such as an aliphatic carboxylic acid ester of lower boiling point, e.g., below 200°C, or higher boiling point, e.g., below 250°C. The aliphatic carboxylic acid ester is especially an
5 aliphatic dicarboxylic acid dilower alkyl ester or ester mixture, typically an alkanedioic acid dilower alkyl ester (lower alkyl alkanedioate) or ester mixture, such as succinic, glutaric and adipic acid dimethyl ester, or a succinic, glutaric and adipic
10 acid dilower alkyl ester mixture such as 10-20% dilower alkyl succinate, 60-70% dilower alkyl glutarate and 10-20% dilower alkyl adipate, and, particularly, a mixture of refined methyl diesters of succinic, glutaric and adipic acids, or other such
15 volatilizable ester in which the flux contents are soluble.

Such a preferred mixture of refined methyl diesters is sold under the trade designation "DBE" solvent (DuPont Co.), as a liquid of about average
20 molecular weight (m.wt.) 159, 5.3% solubility in water (20°C), flashpoint 100°C, viscosity 2.4 cst. (25°C), and surface tension 35.6 dynes/cm (20°C), which has a boiling point of bp 196-225°C and a specific gravity of 1.092 (20°C), and contains 99.5% total ester
25 content (min.), comprising of about 16.5% dimethyl succinate, 66% dimethyl glutarate and 17% dimethyl adipate, plus approximately 0.2% methanol, 0.1% water (max.) and less than 10 ppm hydrogen cyanide.

Other such refined methyl diesters or
30 diester mixtures are those sold under the following trade designations (DuPont Co):

"DBE-2" solvent, as a liquid of about

average molecular weight of 163, 4.2% solubility in water (20°C), flash point 104°C, and viscosity 2.5 cst. (25°C), which has a boiling point of 210-225°C and a specific gravity of 1.081 (20°C), and
5 contains 99.5% total ester content (min.), comprising of about 0.5% dimethyl succinate, 76% dimethyl glutarate and 23% dimethyl adipate, plus approximately 0.1% methanol and 0.1% water (max.).

"DBE-3" solvent, as a liquid of about
10 average molecular weight of 173, 2.5% solubility in water (20°C), flash point 102°C, and viscosity 2.5 cst. (25°C), which has a boiling point of 215-225°C and a specific gravity of 1.068 (20°C), and contains 99.5% total ester content (min.), comprising
15 of about 0.5% dimethyl succinate, 10% dimethyl glutarate and 89% dimethyl adipate, plus approximately 0.1% methanol and 0.3% water (max.).

"DBE-4" solvent, as a liquid of about
average molecular weight 146, 7.5% solubility in water
20 (20°C), flash point 94°C, and viscosity 2.5 cst. (25°C), which has a boiling point of about 196°C and a specific gravity of 1.121 (20°C), and contains 99.5% total ester content (min.), comprising of about 99.5+% dimethyl succinate, plus approximately 0.1% methanol
25 and 0.1% water (max.).

"DBE-5" solvent, as a liquid of about
average molecular weight 160, 4.3% solubility in water
(20°C), flash point 107°C, and viscosity 2.5 cst.
(25°C), which has a boiling point of 210-215°C and a
30 specific gravity of 1.091 (20°C), and contains 99.5% total ester content (min.), comprising of about 0.6% dimethyl succinate, 99% dimethyl glutarate and 0.1%

dimethyl adipate, plus approximately 0.1% methanol and 0.1% water (max.).

"DBE-9" solvent, as a liquid of about average molecular weight 156, 5% solubility in water (20°C), flash point 94°C, and viscosity 2.4 cst. (25°C), which has a boiling point of 196-215°C and a specific gravity of 1.099 (20°C), and contains 98.5% total ester content (min.), comprising of about 25% dimethyl succinate, 73% dimethyl glutarate and 1.5% dimethyl adipate, plus approximately 0.3% methanol and 0.5% water (max.).

The aliphatic carboxylic acid ester, e.g., where it has a boiling point of from 196°C to above 200°C, is preferably used in admixture with an above type alcohol to lower the solvent boiling point.

Similarly, the solvent may be a cyclic ketone such as a cycloaliphatic, including azacycloaliphatic (i.e., N-heterocyclic), ketone, e.g., cyclopentanone, cyclohexanone, N-methyl-2-pyrrolidone (about bp 202°C), or other such volatilizable ketone in which the flux contents are soluble. The cyclic ketone may also be used in admixture with an above type alcohol and/or ester to lower the solvent boiling point.

Whilst the rosin flux is typically in liquid form, it may also be in paste or solid form, by admixing with a suitable volatilizable paste or solid carrier vehicle or extender, e.g., liquid or jelly (paste form) glycerol or a wax (solid form). All carrier vehicles or extenders used to provide the liquid, paste or solid flux must be chemically inert and compatible with the flux contents, solder and

metal surfaces to be soldered, and capable of volatilizing during soldering to leave the soldering site essentially ionic residue-free.

In the rosin flux, the solvent is
5 preferably miscible with water to enable water to be added optionally as a diluent to decrease solvent cost and enhance foamability of the flux, thereby aiding its spreading action. As the solvent and water boil off during soldering, their presence is
10 not detrimental to the product. No more than a minor amount of water is used so as to inhibit splattering and deposition of residue at the soldering site, and possibly solder ball formation, that adversely affect product quality. As an essentially ionic
15 residue-free product is desired, de-ionized water is used to avoid increasing the ionic residue.

In heat-soldering metal surfaces, solder is applied to the surfaces in the presence of an effective amount of the flux in coating disposition
20 on the surfaces, and at a temperature sufficient for molten flow of the solder and for volatilizing the

flux to leave the site essentially ionic residue-free. The soldering may be at conventional temperature, e.g., 200-500°C, preferably 250-350°C. For example, circuit board components may be soldered at the lower
5 end of the temperature range and other connections at the higher end of the range.

The solder may be of any known type such as tin-lead solder, e.g., 60% Sn - 40% Pb of mp 191-193°C, or eutectic tin-lead solder, e.g.,
10 63% Sn - 37% Pb of mp 183°C.

The flux is applied to the surfaces to be joined by conventional techniques, e.g., foaming, wave fluxing, spraying, brushing, rolling, and dipping, and the solder is applied to the flux-coated surfaces in
15 usual manner, e.g., drop dispensing, or wave soldering, to form the solder deposit. Then, the soldered surfaces are contacted with one another to join them at the soldering temperature, which also causes boiling-off of the flux constituents.

20 Normally, the attaining of a low ionic residue at the site is a function of the soldering temperature used during the operation. Thus, the amount of the three-acid mixture in the rosin flux is selected to achieve adequate activation of the rosin
25 and essentially complete volatilization of the three-acid mixture under the given soldering conditions to assure that only a low ionic residue will remain.

Simple test runs may be used to optimize the
30 three-acid mixture amount needed in the flux for the

intended purposes at the given soldering temperature whilst also satisfying the aim of leaving a low ionic residue at the soldering site.

In sum, the invention provides a
5 non-corrosive flux for soldering metals (e.g., tin plate, copper, brass, nickel, etc.), wherein the three-acid mixture imparts properties to the flux significantly different from those of the individual acids, for adequate fluxing action, yet leaving
10 minimal ionic residue.

Particular advantages of the flux of the invention include:

(1) A three-acid mixture maximum boiling range, e.g., 150-210°C, that is significantly below
15 the 235°C boiling point of succinic acid, its component of lowest boiling temperature.

(2) A three-acid mixture which does not sublime like its component succinic acid, and thus does not condense on adjacent areas, e.g., of circuit
20 boards, during soldering, even though adequate precaution is not taken to minimize this effect.

(3) A three-acid mixture of unique quality permitting use of rosin fluxes of as low as 1-2% rosin content providing adequate foaming during soldering
25 without adding a surfactant.

(4) A composition of pleasant odour and that does not form noxious and pungent break-down products during soldering.

(5) A three-acid mixture that costs less
30 than each of its constituents that could be used individually as an activator.

(6) The attaining of the above-listed advantages with a three-acid mixture as essential component, whether in purified state or not, and thus contemplating commercial purity three-acid mixtures, whilst providing a stable flux of good cleaning action leaving so low a residue after soldering that no corrosive property is exhibited and no residue removal is needed.

The proportions of the three acids to each other in the three-acid mixture, and of all components in the flux, are selected to provide surface tension properties and a boiling point that enhance flux spreading and "wetting" of the surfaces being soldered and volatilization with innocuous decomposition without splattering, subliming or local re-deposition.

At most, only a trace amount of residue is left on the product. The ionic content of this residue, expressed as micrograms per square centimetre equivalent of NaCl (per Omega (RTM) Meter measurement, as later explained), is very low, i.e., below about $5 \mu\text{g}/\text{cm}^2$, and especially below about $3 \mu\text{g}/\text{cm}^2$ for the rosin flux.

As used herein, the term a "trace" amount means a very small amount that is less than about $5 \mu\text{g}/\text{cm}^2$, i.e., a microgram amount corresponding to less than five millionths of a gram (0.000,001 g) per sq. cm. area (cf. Hackh's Chemical Dictionary, 4th ed. 1969; p. 686: "trace" - a very small quantity, usually less than 5 gamma per gm; p. 288:

"gamma" - 10^{-6} gm; p. 429: "microgram" - μg , gamma, one-millionth of a gram).

On using the flux of the invention, cleanliness tests for residual ionic contamination (per said Omega (RTM) Meter measurement) showed that unwashed soldered electronic circuit boards had ionic residues below a trace amount, while corresponding soldered boards after washing in Freon TMS (DuPont Co.), a chlorinated solvent, had only slightly lower residues. Thus, use of the flux of the invention affords a reliable quality product of acceptably low ionic residue without cleaning, and, even with cleaning, the ionic residue is only slightly further reduced.

The treatise, Kirk-Othmer Encyclopedia of Chemical Technology, 3rd ed., John Wiley & Sons, provides a review of "Dicarboxylic Acids" (vol. 7: 614-625), i.e., the saturated, linear aliphatic series of dicarboxylic acids (diacids), from which melting and boiling point data as to the first nine of these organic dibasic acids are compiled in Table A.

TABLE A - Melting and Boiling Points

Total carbon atoms	UIPAC name	Common name	mp °C	bp °C
2	ethanedioic	oxalic	187 (dec)	
3	propanedioic	malonic	134-136 (dec)	
4	butanedioic	succinic	187.6-187.9	
5	pentanedioic	glutaric	98-99	200 2.7 kPa
6	hexanedioic	adipic	153.0-153.1	265 13.3 kPa
7	heptanedioic	pimelic	105.7-105.8	272 13.3 kPa
8	octanedioic	suberic	143.0-143.3	279 13.3 kPa
9	nonanedioic	azelaic	107-108	286.5 13.3 kPa
10	decanedioic	sebacic	134.0-134.4	294.5 13.3 kPa

*To convert kPa to mm Hg, multiply by 7.5

2.7 kPa x 7.5 = 20 mm Hg; 13.3 kPa x 7.5 = 100 mm Hg.

From that same treatise, data as to the ionization

constants of these diacids are also compiled in Table B.

TABLE B - Ionization Constants in Water

Diacid	Temp., °C	K	
		K1	K2
oxalic	25	5.36×10^{-2}	5.42×10^{-5}
malonic	25	1.42×10^{-3}	2.01×10^{-6}
succinic	25	6.21×10^{-5}	2.31×10^{-6}
glutaric	25	4.58×10^{-5}	3.89×10^{-6}
adipic	25	3.85×10^{-5}	3.89×10^{-6}
pimelic	18	3.19×10^{-5}	3.74×10^{-6}
suberic	18	3.05×10^{-5}	3.85×10^{-6}
azelaic	18	2.88×10^{-5}	3.86×10^{-6}
sebacic	25	3.1×10^{-5}	3.6×10^{-6}

Selected characteristics based on Tables A and B,
and the molecular weights of these diacids are
given in Table C.

TABLE C - Selected Characteristics

Acid	Formula	M.Wt.	mp °C	bp °C	K1
oxalic	HOOC - COOH	90	187d		5.36×10^{-2}
malonic	HOOC CH ₂ COOH	104	135d		1.42×10^{-3}
succinic	HOOC(CH ₂) ₂ COOH	118	188	200*	6.21×10^{-5}
glutaric	HOOC(CH ₂) ₃ COOH	132	98.5	265**	4.58×10^{-5}
adipic	HOOC(CH ₂) ₄ COOH	146	153	272**	3.85×10^{-5}
pimelic	HOOC(CH ₂) ₅ COOH	160	106	279**	3.19×10^{-5}
suberic	HOOC(CH ₂) ₆ COOH	174	143	286.5**	3.05×10^{-5}
azelaic	HOOC(CH ₂) ₇ COOH	188	107.5	294.5**	2.88×10^{-5}
sebacic	HOOC(CH ₂) ₈ COOH	202	134		3.1×10^{-5}

* 20 mm Hg

**100 mm Hg

That treatise points out that these diacids are crystalline solids, whose melting points decrease as the aliphatic chain length (m. wt.) increases, with alternation of some physical properties of neighbouring members of the series such as oscillating melting point, water solubility and ease of decarboxylation or decomposition, but not of others such as boiling point, density and dielectric constant. Whereas the odd-numbered acids melt lower than their neighbours, e.g., the 5 carbon atom glutaric acid melting much lower than the neighbouring 4 carbon atom succinic acid and 6 carbon atom adipic acid, their boiling points increase with increasing molecular weight.

The boiling points of the lower (m. wt.) diacids are difficult to measure as they sublime, dehydrate or decompose, the even-numbered carbon atom acids being more resistant to decarboxylation or decomposition than the odd-numbered acids preceding them. Their thermal decomposition is sensitive to catalytic effects of impurities, environment and the decomposition products themselves, and results in decarboxylation to a monocarboxylic acid or anhydride formation, with water elimination, succinic and glutaric acids forming cyclic anhydrides and adipic acid forming a polymeric (linear) anhydride.

Malonic and glutaric acids are extremely water-soluble, the remaining even-numbered acids being only moderately soluble, solubility alternating between higher values for the odd-numbered acids and lower values for their even-numbered neighbours, yet rapidly decreasing with increasing chain length (m. wt.). Oxalic and malonic acids are strong acids

and the higher homologues (m. wt.) are progressively weaker. Whilst the odd-numbered acids have lower ionization constants than their even-numbered neighbours, the ionization constant values become
5 almost constant for all the acids above adipic acid.

Elsewhere, that treatise notes that malonic acid loses carbon dioxide on heating above its melting point, and in aqueous solution, undergoes decarboxylation at 70°C (vol. 14: 794-795); that
10 succinic acid sublimates when heated below its 188°C melting point, dehydrates at its melting point, and forms a cyclic anhydride, succinic anhydride having mp 119.6°C (vol. 21: 848-849); and that adipic acid, unlike glutaric acid and succinic acid, does not
15 readily form a cyclic anhydride, but decomposes to cyclopentanone above 225°C (vol. 1: 510-513).

The Merck Index, 10th ed. 1983, Merck & Co., Rahway, N.J., shows similar data as to these nine diacids, especially their boiling points at
20 normal pressure (760 mm Hg). It indicates that oxalic acid sublimates at 157°C, decomposes at higher temperature, and is hygroscopic and poisonous; that malonic acid melts with decomposition at about 135°C; that succinic acid has mp 185-187°C and bp 235°C with
25 partial conversion to the anhydride (of mp 119.6°C and bp 261°C); that glutaric acid has mp 97.5-98°C and bp 302-304°C with very slight decomposition; that adipic acid has mp 152°C and bp 337.5°C, and is not hygroscopic; and that pimelic acid tends to sublime.

30 Oxalic and malonic acids are undesirable for this invention because of their low molecular weights, strong acidity and high dissociation constants, and the fact that oxalic acid sublimates at

157°C and is hygroscopic whilst malonic acid decomposes at its 135°C melting point, thereby adversely affecting the flux characteristics and causing large ionic residues, e.g., from
5 decomposition and sublimation products.

Pimelic and higher acids are undesirable for this invention because of their cost, and high molecular weights and boiling points (which increase with their increasing molecular weights) that may
10 adversely affect the characteristics and adequacy of boiling-off of the flux. Pimelic acid is not only costly, but also may splatter and sublime to expose the circuit board to local deposition of sublimation or decomposition constituents detracting from product
15 quality.

Whilst succinic acid also tends to sublime, and glutaric acid is quite water-soluble, compared to non-hygroscopic adipic acid, on using these three acids as a mixture in the above-stated proportions,
20 the average molecular weight and depressed boiling point of the mixture, as opposed to the individual acids, seem to impart efficient spreading action to the flux, and the higher content of the lower boiling point glutaric acid seems to aid boiling-off of the
25 lower content higher boiling point succinic and adipic acids, as it has been found that the flux spreads without voids, does not splatter and leaves a low residue.

The rosin for the rosin flux of the
30 invention may be water-white rosin or rosin in suitable formulation form, such as a commercially available liquid, e.g., a solution in a volatile solvent such as isopropyl alcohol, or solid, e.g., a synthetic rosin-containing resin preparation.

A first commercial rosin formulation, sold under the trade designation KESTER no. 135 Flux (Litton/Kester Solder Co., Des Plaines, IL), as a non-activated, Type R, rosin flux, is a homogeneous solution of water-white rosin in isopropyl alcohol, of non-corrosive and non-conductive properties like rosin, and processed to remove the foreign particles found in natural rosin. It has a composition of 41% rosin plus 59% isopropyl alcohol, 77°C (170°F) boiling point, sp.g. 0.880, 66 vol.% volatile content, 59% water solubility and 18°C (65°F) flash point.

A second commercial rosin formulation, sold under the trade designation ULTRA-DRY Flux no.UD-3040 (Urban Chemical Co., Deerfield, IL), has a composition of 40% rosin and 60% isopropyl alcohol, 93°C (200°F) boiling point, sp.g. 0.884, 66 vol.% volatile content and 21°C (70°F) flash point.

A third commercial rosin formulation, sold under the trade designation STAYBELITE (RTM) resin (Hercules Inc., Wilmington, DE), is a synthetic rosin-containing resin preparation, comprising a solid product formed of hydrogenated rosin.

The three-acid mixture or TAM used for the rosin flux of the invention may also be from any source, such as a commercial mixture of succinic, glutaric and adipic acids in which the three acids are in the desired proportions.

A first commercial mixture is a blend of the three acids available from DuPont Canada, Inc. (DuPont Canada), of good solubility, mild acidity (pH 2.95), melting range 90-130°C and boiling range 170-195°C, which has:

TAM Formulation no. 1 - assay 97.0% (min.), 26-30% succinic acid, 55-59% glutaric acid and 11-15% adipic acid, plus 3% acid anhydrides (average), 0.8% succinimide (max.), 0.6% glutarimide (max.), 0.3% water (max.), 1 ppm copper (max.), 1 ppm vanadium (max.) and 5 ppm iron (max.). Typically, it has an assay of 97.6%, 27.7% succinic acid, 56.6% glutaric acid and 13.2% adipic acid, plus 2.7% acid anhydrides, 0.7% succinimide, 0.56% glutarimide, 0.08% water, 0.28 ppm copper, 0.1 ppm vanadium and 1.9 ppm iron; or it has about 27% succinic acid, 57.5% glutaric acid and 11.6% adipic acid, plus about 4% acid anhydrides, 0.7% succinimide, 0.5% glutarimide and 0.2% water.

A second commercial mixture is the product, designated "DBA", available from DuPont Co., Wilmington, DE (DuPont), of 20% water solubility (18°C/64°F), which has:

TAM Formulation no. 2 - 18-28% succinic acid, 51-61% glutaric acid and 15-25% adipic acid, plus 1% organic nitrogen compounds, 0.2% nitric acid, 0.02% copper and 0.01% vanadium, and is available in a 50% concentration solution of the three acids in de-ionized water, sp.g. 1.106, having 26% succinic acid, 55% glutaric acid and 18% adipic acid, plus 0.3% nitric acid.

A third commercial mixture is a dicarboxylic acid product available from BASF Corp., Parsippany, NJ, of about 10% water solubility (room temp.), and 100-130°C melting range and 150-210°C boiling range, which has:

TAM Formulation no. 3 - assay of 97% (min.), 25-30% succinic acid, 42-47% glutaric acid and 25-30%

adipic acid, plus 2.3% 2,5-pyrrolidinedione and 2,6-piperidinedione, and 0.5% water.

A fourth commercial mixture is the product, designated "Diacides AGS", available from
5 Rhone-Poulenc Inc., Princeton, NJ, of about 10% water solubility (room temp.) and 100-130°C melting range, which has:

TAM Formulation no. 4 - 18-25% succinic acid, 50-55% glutaric acid and 20-25% adipic acid,
10 plus 1.4% succinic anhydride, and particularly 19.5% succinic acid, 51.6% glutaric acid and 22.6% adipic acid, plus 1.4% acid anhydrides (as succinic anhydride), 0.06% nitrogen (kjeldahl), 0.3% water, 0.4 ppm copper, 0.06 ppm vanadium and 2.4 ppm iron.

15 These TAM formulations are especially usable as the three-acid mixture, with the components in the range of 18-30% succinic acid, 51-61% glutaric acid and 11-30% adipic acid.

It was quite unexpected that these
20 three-acid mixtures, per TAM formulations no.1-4, which are not especially purified, perform satisfactorily in the flux to provide consistent solderability under the relatively rigid conditions extant in electronic circuit board component
25 soldering, yet leave the site essentially free from ionic residue and avoid a washing step, despite the impurities in such commercial grade mixtures.

This is especially so in view of the above indication in the Kirk-Othmer treatise that thermal
30 decomposition of these diacids is sensitive to catalytic effects of impurities, and potentially those listed in commercial TAM formulations no. 1-4.

The costs of chemicals in pure and

commercial grade form are influenced by many factors, yet provide useful comparisons. Recently noted costs of the three pertinent acids in pure form, and the next higher acid, pimelic, are compared in Table D.

5

TABLE D - Comparative Costs of Individual Acids

	<u>Acid</u>	<u>Unit Cost</u>	<u>Calculated Cost Per Kg</u>	
10	Succinic	\$126.90/2.5kg	\$ 50.76	50.76
	Glutaric	14.20/25g	568.00	-
	Adipic	24.60/2kg	12.60	12.60
	Pimelic	61.25/100g	-	<u>612.50</u>
	Total		631.36	675.86
15	Average		210.45	225.28

Arbitrarily taking equal amounts of the three acids as a mixture, the cost comes to an average cost of \$210.45/kg, and substituting pimelic for glutaric acid increases it to \$225.28/kg, and even higher if it is substituted for succinic or adipic acid. Any such substitution would change the properties of the resulting mixture, given the differences in melting and boiling points among these acids and the increase in average molecular weight imparted by the heavier pimelic acid component, aside from the objectionable tendency of pimelic acid to sublime.

More important, the cost of the commercial purity mixtures of succinic, glutaric and adipic acids, per TAM formulations no. 1-4 above, is roughly \$1.25/kg (\$0.57/lb), which is far less than the above

30

calculated average cost of a mixture of the pure acids, and a comparable mixture in which pimelic acid is substituted for glutaric acid would be even more expensive. Despite the lower purity of these commercial mixtures in the flux and the need to limit the ionic residue to, at most, a trace amount, they surprisingly perform eminently satisfactorily for the purposes of the invention, without having to formulate the mixture from the high purity individual acids at high cost.

Based on the above Merck Index data, their melting and boiling points may be taken generally as set forth in Table E.

TABLE E - Approximate Melting and Boiling Points

<u>Acid</u>	<u>mp °C</u>	<u>bp °C</u>
Succinic	186	23
Glutaric	98	303
Adipic	<u>152</u>	<u>337.5</u>
Total	436	875.5
Average	145°C	292°C

The commercial mixtures of these acids have 90-130 and 100-130°C mp ranges, and 170-195 and 150-210°C bp ranges, of average mp 110°C and bp 180°C well below the Table E averages. The commercial mixture mp range is below the boiling points of succinic and adipic acid, and its bp range is below the boiling point of succinic acid, i.e., the lowest of the three acids.

A distinct benefit of the commercial mixture depressed boiling range is that the acids boil away to leave a low ionic residue, so that the impurities in the commercial grade mixtures and high acidity of their dibasic acid content have been found not to be a problem. When the three acids are in the above-noted ranges, optimum results are achievable for adequately coating the surfaces to be soldered, whilst inhibiting splattering, sublimation and local re-deposition.

Despite the high acid content and impurities in the commercial mixtures, the constituents boil off at ambient soldering conditions, avoiding a large ionic residue and a washing step for its removal, and the low ionic residue that is left occurs without supplemental heating as normally used to prevent local re-deposition of constituents. The flux assures more consistent solderability in attaining a low ionic residue product, and soldering occurs without causing annoying or noxious odours on the circuit boards or fixtures, and provides a circuit board product whose surfaces have a desired post-solder dryness and non-tack condition without use of special steps.

Recently noted costs of pertinent earlier mentioned commercially available chemical and chemical formulation components of the flux are compared in Table F.

TABLE F - Comparative Costs of Various Components

<u>Component</u>	<u>Cost Per Gal.</u>	<u>Calculated Cost Per Kg</u>
DBE solvent	\$5.15-5.20	\$1.25-1.26
KESTER #135	10.70	3.21
(41% rosin)	-	(1.32)
(59% IPA)	-	(1.89)
ULTRA-DRY Flux		
#UD-3040	19.55	5.84
(40% rosin)	-	(2.34)
(60% IPA)	-	(3.50)
STAYBELITE		
resin	-	2.75-2.97*
IPA	2.19-2.20	0.74-0.75

* 1.25-1.35/lb

Table F shows that the cost of "DBE" solvent (sp.g. 1.092) is about the same as the above-indicated cost of the TAM formulations nos. 1-4, and less than the calculated, arbitrarily 41% or 40% allocated, rosin portion cost of the respective commercial IPA (isopropyl alcohol) solutions of rosin (KESTER no. 135, sp.g. 0.880; ULTRA-DRY Flux no. UD-3040, sp.g. 0.884), as well as less than the cost of the hydrogenated rosin preparation (STAYBELITE resin). Given the low cost of IPA (sp.g. 0.78), the cost of the rosin portion of the commercial IPA solutions of rosin (KESTER no. 135 and ULTRA-DRY Flux no. UD-3040) must actually be considered higher, and its IPA portion cost lower, than as shown in Table F.

The rosin flux of the invention is of advantage, as it has an acceptably low materials cost, and achieves an acceptably low ionic content flux residue on soldering. Most significant is the fact that the rosin flux can provide a soldered product in usable form as produced, without the trouble and expense of a washing step to remove ionic residue and/or of supplying supplemental heat during the soldering to assist volatilization of the flux constituents.

Typically, the rosin flux is useful in soldering output assemblies to copper buffer plates on ignition assemblies of motor vehicles, and the rosin flux is useful in electronic circuit board soldering to attain in all

cases a significant decrease in the number of product rejects from shorts, voids and like defects, without a washing step.

5 However, in using the rosin flux for
circuit board soldering, there may be instances when
efficient electrical contact is difficult to achieve
between certain mechanical switch contact surfaces
(e.g., on gold or silicone rubber mechanical switch
10 components), due to the interfering presence of a
slight residue of rosin that acts as an electrical
insulator thereat. This can occur even though the
attendant ionic residue is acceptably low.

 As the optional inclusion of de-ionized
water further lowers the cost and aids foaming where
15 desired, its hydrolyzing effect on the ester
component can be readily controlled by conjoint
inclusion of a hydrolysis inhibiting agent. The
influence of the low surface tension property of the
ester component on the alcohol component and included
20 water will thereby be retained, whilst any increase
in acidity of the three-acid mixture-containing
solution, traceable to ester hydrolysis, will be
minimized.

 The following examples in which all parts
25 and percentages are by weight unless otherwise

indicated, are set forth by way of illustration and not limitation of the invention claimed.

EXAMPLE 1 - High Rosin Aqueous Alcoholic Flux

5 (a) The above-noted TAM (three-acid mixture) Formulation no. 1 (DuPont Canada), of 26-30% succinic acid, 55-59% glutaric acid and 11-15% adipic acid, plus 3.0% acid anhydrides, 0.8% succinimide, 0.6% glutarimide and 0.3% water, was dissolved
10 in de-ionized water in proportions to form an aqueous TAM solution of sp.g. 1.084 (0.3703703 g/ml TAM and 0.7136296 g/ml water, total 1.084 g/ml), corresponding to 135 ml of 50 g TAM (34.2%) and 96.34 g water (65.8%), total 146.34 g; or to 1401.29 ml
15 of 519 g TAM (34.2%) and 1000 g water (65.8%), total 1519 g.

(b) 2705 ml of KESTER no.135 Flux solution, sp.g. 0.88, totalling 2380.4 g (0.88×2705), of 41% or 975.96 g (2380.4×0.41) rosin and 59% or 1404.44 g (2380.4×0.59) IPA (isopropyl alcohol), were
20 combined with 780 ml or 845.52 g (780×1.084) TAM solution from (a), of 288.89 g (780×0.3703703) TAM (34.2%) and 556.63 g (780×0.7136296) water (65.8%), plus 300 ml IPA (sp.g. 0.78), or 234 g (300×0.78),
25 to form 3785 ml or 1 U.S. gal. of flux.

The flux, by calculation, contained 987.96 g rosin (28.2%), 288.89 g TAM (8.3%), 1638.44 g ($1404.44 + 234$) IPA (47.4%) and 556.63 g water (16.1%), total 3459.92 g of sp.g. 0.9141
30 ($3459.92/3785$), for a rosin content of 28.2% corresponding to 26 vol.% ($987.96/3785$).

EXAMPLE 2 - High Rosin Aqueous Alcoholic Flux

EXAMPLE 1 was repeated except that 3095 ml of KESTER no. 135 Flux solution, totalling 2723.6 g (0.88 x 3095), of 41% or 1116.68 g (2723.6 x 0.41) rosin and 59% or 1606.92 g (2723.6 x 0.59) IPA, were combined with 390 ml or 422.76 g (390 x 1.084) TAM solution, of 144.44 g (390 x 0.3703703) TAM (34.2%) and 278.32 g (390 x 0.7136296) water (65.8%), plus 300 ml or 234 g IPA, to form 3785 ml or 1 U.S. gal. of flux.

The flux, by calculation, contained 1116.68 g rosin (33.0%), 144.44 g TAM (4.3%), 1840.92 g (1606.92 + 234) IPA (54.5%) and 278.32 g water (8.2%), total 3380.36 g of sp.g. 0.8931 (3380.36/3785), for a rosin content of 33.0% corresponding to 30 vol.% (1116.68/3785).

EXAMPLE 3 - High Rosin Aqueous Alcoholic Flux

(a) The above-noted TAM Formulation no.2 in a 50% concentration solution of the three acids in de-ionized water (DuPont), sp.g. 1.106, of 26% succinic acid, 55% glutaric acid and 18% adipic acid, plus 0.3% nitric acid, was used in (b). By calculation, this 50/50 concentration solution corresponded to 0.553 g/ml TAM and 0.553 g/ml water, total 1.106 g/ml.

(b) EXAMPLE 1 was repeated except that the 50% concentration TAM solution of (a) was used as the TAM solution, and 2765 ml of KESTER no. 135 Flux solution, totalling 2433.2 g (0.88 x 2765), of 41% or 997.61 g (2433.2 x 0.41) rosin and 59% or 1435.59 g (2433.2 x 0.59) IPA, were combined with 720 ml or 796.32 g (720 x 1.106) TAM solution, of 398.16 g

(720 x 0.553) TAM (50%) and 398.16 g (720 x 0.553) water (50%), plus 300 ml or 234 g IPA, to form 3785 ml or 1 U.S. gal. of flux.

5 The flux, by calculation, contained 997.61 g rosin (28.8%), 398.16 g TAM (11.5%), 1669.59 g (1435.59 + 234) IPA (48.2%) and 398.16 g water (11.5%), total 3463.52 g of sp.g. 0.9151 (3463.52/3785), for a rosin content of 28.8% corresponding to 26 vol.% (997.61/3785).

10

EXAMPLE 4 - High Rosin Aqueous Alcoholic Flux

EXAMPLE 3 was repeated except that 2705 ml of KESTER no. 135 Flux solution, totalling 2380.4 g (0.88 x 2705), of 41% or 975.96 g (2380.4 x 0.41) 15 rosin and 59% or 1404.44 g (2380.4 x 0.59) IPA, were combined with 780 ml or 862.68 g (780 x 1.106) TAM solution, of 431.34 g (780 x 0.553) TAM (50%) and 431.34 g (780 x 0.553) water (50%), plus 300 ml or 234 g IPA, to form 3785 ml or 1 U.S. gal. of flux.

20

The flux, by calculation, contained 975.96 g rosin (28.1%), 431.34 g TAM (12.4%), 1638.44 g (1404.44 + 234) IPA (47.1%) and 431.34 g water (12.4%), total 3477.08 g of sp.g. 0.9186 (3477.08/3785), for a rosin content of 28.1% 25 corresponding to 26 vol.% (975.96/3785).

Table G shows the proportions (gms, % and parts) of the ingredients in the high rosin content fluxes of Examples 1-4.

Table G shows that the fluxes of EXAMPLES 1-4 have 28-33% rosin, 4-13% TAM, 47-55% IPA and 8-16% water, or 36-41% active and 59-63% other components. At 100 pts rosin, they have 13-44 pts TAM, 165-168 pts IPA and 25-57 pts water.

These fluxes are used in general metal soldering, e.g., of copper, brass, nickel and tin surfaces, typified in EXAMPLE 5.

10 EXAMPLE 5 - General Metal Soldering.

An aluminium backplate (66.77 sq. cm. (710.35 sq. in.) area), provided with two copper slugs (11.94 mm x 8.13 mm x 0.813 mm (0.47" x 0.32" x 0.032") strips), was placed on a walking beam (a support) and the flux of EXAMPLE 1 then drop-dispensed onto the slugs so that the flux spread over their entire surfaces. The flux was also drop-dispensed onto the surfaces of a strip of tin-lead solder (60% Sn - 40% Pb; mp 191-193°C) which was then placed on top of the flux-coated slugs. The entire assembly was then pre-heated at 180°C for 86 seconds.

Following pre-heating, an electroless-plated or electroplated nickel substrate on a beryllium oxide substrate, of similar dimensions to the copper slugs, onto which nickel substrate a Darlington transistor had been earlier soldered by die attachment in ambient hydrogen at 400-440°C, was placed on the flux-coated solder on the backplate, and the assembly heated at 230-250°C to cause solder flow and joining of the components, after which the assembly was cooled. The nickel substrate had a thin oxide layer, and where it was electroless-plated

nickel, it had a phosphorus oxide layer formed as a hard, tenacious oxide, that required strong fluxing action for proper soldering.

5 The soldered part was not cleaned in trichlorethylene as is needed to clean excess of the currently used known flux no. 1 (KESTER 1544, an activated 50% rosin flux of undisclosed proprietary formulation) from the backplate. Experience has shown that such excess of known flux no.1 contributes to electrical leakage of the Darlington transistor, yet the EXAMPLE 1 flux left so low a residue that this leakage was reduced to an inconsequential level.

10 This beneficial result was unexpected, considering the soldering temperature and high amount of diacids in the TAM used to activate the rosin, as shown in Table G, for cleaning and de-oxidizing, e.g., of tenaciously held oxide layers on the copper and nickel surfaces, in non-circuit board electrical connection soldering, where splattering is not as important a consideration as with circuit board soldering. Whilst known flux no. 1 is of higher rosin content than the flux of the invention, the leakage is clearly not caused by rosin, but by the high ionic residue traceable to the high ionic content in the known flux.

25 The above soldering procedure was repeated with each of the fluxes of EXAMPLES 2-4, and the same low ionic residue beneficial results were obtained in each case.

30

EXAMPLE 6 - Low Rosin Aqueous Alcoholic Flux

(a) The above-noted TAM Formulation no. 3 (BASF) of at least 97% purity, 25-30% succinic acid,

42-47% glutaric acid and 25-30% adipic acid, plus 2-3% total 2,5-pyrrolidinedione and 2,6-piperidinedione, was dissolved in a 60% IPA solution in de-ionized water in proportions to form
 5 an aqueous alcoholic TAM solution of sp.g. 0.9796 (0.2815 g/ml TAM, 0.3764 g/ml IPA and 0.3217 g/ml water, total 0.9796 g/ml), corresponding to 525 g TAM in 1500 ml of the 60% solution of 900 ml or 702 g IPA (0.78 x 900) and 600 ml or 600 g water, to form
 10 1865 ml TAM solution of 525 g TAM (28.7%), 702 g IPA (38.4%) and 600 g water (32.9%), total 1827 g.

(b) 525 ml of ULTRA-DRY Flux no. UD-3040 solution, sp.g. 0.884, totalling 464.1 g (0.884 x 525), of 40% or 185.64 g (525 x 0.40) rosin and 60%
 15 or 278.46 g (525 x 0.60) IPA, were combined with 155 ml or 151.83 g (155 x 0.9796) TAM solution from (a), of 43.63 g (155 x 0.2815) TAM (28.7%), 58.34 g (155 x 0.3764) IPA (38.4%) and 49.86 g (155 x 0.3217) water (32.9%), plus 8782 ml or 6849.90 g
 20 (0.78 x 8782) IPA, to form 9462 ml or 2.5 U.S. gal. of flux.

The flux, by calculation, contained 185.64 g rosin (2.5%), 43.63 g TAM (0.6%), 7186.70 g (6849.90 + 278.46 + 58.34) IPA (96.2%) and 49.86 g
 25 water (0.7%), total 7465.89 g of sp.g. 0.7890 (7465.89/9462), for a rosin content of 2.5% corresponding to 2 vol.% (185.64/9462).

(c) The procedures of (a) and (b) were repeated using instead the above-noted TAM
 30 Formulation no. 4 (Rhone-Poulenc), of 18-25% succinic acid, 50-55% glutaric acid and 20-25% adipic acid, plus 1.4% succinic anhydride.

(d) The procedures of (a) and (b) were repeated using instead TAM Formulation no. 1 (DuPont Canada) of EXAMPLE 1.

5 **EXAMPLE 7 - Low Rosin Aqueous Alcoholic Flux**

(a) The procedures of EXAMPLE 6 (a) and (b) were repeated, this time using 560 ml or 495.04 g (0.884×560) of ULTRA-DRY Flux no. UD-3040 solution, of 40% or 198.02 g (495.04×0.40) rosin and 60% or 297.02 g (495.04×0.60) IPA, with 165 ml or 161.64 g (165×0.9796) TAM solution, of 46.45 g (165×0.2815) TAM (28.7%), 62.11 g (165×0.3764) IPA (38.4%) and 53.08 g (165×0.3217) water (32.9%), plus 8737 ml or 6814.86 g (0.78×8737) IPA, to form 9462 ml or 2.5 U.S. gal. of flux.

The flux, by calculation, contained 198.02 g rosin (2.7%), 46.45 g TAM (0.6%), 7173.99 g ($6814.86 + 297.02 + 62.11$) IPA (96.0%) and 53.08 g water (0.7%), total 7471.54 g of sp.g. 0.7896 ($7471.54/9462$), for a rosin content of 2.7% corresponding to 2 vol.% ($198.02/9462$).

(b) The procedure of (a) was repeated using instead TAM Formulation no. 4 (Rhone-Poulenc) of EXAMPLE 6 (c).

25 (c) The procedure of (a) was repeated using instead TAM Formulation no. 1 (DuPont Canada) of EXAMPLE 1.

30 **EXAMPLE 8 - Low Rosin Aqueous Alcoholic Flux**

(a) The procedures of EXAMPLE 6 (a) and (b) were repeated, this time using 425 ml or 375.7 g (0.884×425) of ULTRA-DRY Flux no. UD-3040 solution, of 40% or 150.28 g (375.7×0.40) rosin and 60% or

225.42 g ($375.7 \times .60$) IPA, with 65 ml or 63.68 g
(65×0.9796) TAM solution, of 18.30 g (65×0.2815)
TAM (28.7%), 24.47 g (65×0.3764) IPA (38.4%) and
20.91 g (65×0.3217) water (32.9%), plus 3295 ml or
5 2570.1 g (0.78×3295) IPA, to form 3785 ml or 1
U.S. gal. of flux.

The flux, by calculation, contained 150.28
g rosin (5.0%), 18.30 g TAM (0.6%), 2819.99 g
($2570.1 + 225.42 + 24.47$) IPA (93.7%) and 20.91 g
10 water (0.7%), total 3009.48 g of sp.g. 0.7951
($3009.48/3785$), for a rosin content of 5.0%
corresponding to 4 vol.% ($150.28/3785$).

(b) The procedure of (a) was repeated
using instead TAM Formulation no. 4 (Rhone-Poulenc)
15 of EXAMPLE 6 (c).

(c) The procedure of (a) was repeated
using instead TAM Formulation no. 1 (DuPont Canada)
of EXAMPLE 1.

Table H shows the proportions (gms, % and
20 parts) of the ingredients in the low rosin content
fluxes of Examples 6-8.

TABLE H - Low Rosin Fluxes of the Invention

	Ex. 6	Ex. 7	Ex. 8	
	Grams	Grams	Grams	
	Wt. %	Wt. %	Wt. %	
Rosin	185.64	198.02	150.28	5.0
TAM	43.63	46.45	18.30	0.6
IPA	7186.70	7173.99	2819.99	93.7
Water	49.86	53.08	20.91	0.7
Flux	7465.83	7471.54	3009.48	100
Rosin	185.64	198.02	150.28	89.1
TAM	43.63	46.45	18.30	10.9
Active	229.27	244.47	168.58	100
IPA	7186.70	7173.99	2819.99	99.3
Water	49.86	53.08	20.91	0.7
Other	7236.56	7227.07	2840.90	100
Active	229.27	244.47	168.58	5.6
Other	7236.56	7227.07	2840.90	94.4
Flux	7465.83	7471.54	3009.48	100
Rosin	185.64	198.02	150.28	pts. 100
TAM	43.63	46.45	18.30	pts. 12
IPA	7186.70	7173.99	2819.99	pts. 1876
Water	49.86	53.08	20.91	pts. 14
Sp.G.	0.7890	0.7896	0.7951	
Volume	9462 ml	9462 ml	3785 ml	

Volume 2.5 U.S. gal. 2.5 U.S. gal. 1 U.S. gal.

Table H shows that the fluxes of EXAMPLES 6-8 have 2.5-5% rosin, 0.6% TAM, 93-96% IPA and 0.7% water, or 3-6% active and 94-97% other components. At 100 pts rosin, they have 12-24 pts TAM, 1876-3871 pts IPA and 14-27 pts water.

These fluxes are used in foam or wave-soldering of electronic circuit board components, typified in EXAMPLE 11.

10 EXAMPLE 9 - Low Rosin Water-Free Alcoholic Flux

(a) TAM Formulation no. 3 (BASF) of EXAMPLE 6(a) was used directly by dissolving 15 g thereof in 560 ml or 495.04 g (0.884×560) of ULTRA-DRY Flux no. UD-3040 solution, of 40% or 198.02 g (495.04×0.40) rosin and 60% or 297.02 g (495.04×0.60) IPA, plus 3533 ml or 2755.74 g (0.78×3533) IPA, to form 3785 ml or 1 U.S. gal. of water-free flux.

The flux, by calculation, contained 198.02 g rosin (6.1%), 15 g TAM (0.5%) and 3052.76 g ($2755.74 + 297.02$) IPA (93.4%), total 3265.78 g of sp.g. 0.8628 ($3265.78/3785$), for a rosin content of 6.1% corresponding to 5% vol.% ($198.02/3785$).

(b) The procedure of (a) was repeated using instead TAM Formulation no. 4 (Rhone-Poulenc) of EXAMPLE 6 (c).

(c) The procedure of (a) was repeated using instead TAM Formulation no. 1 (DuPont Canada) of EXAMPLE 1.

30 EXAMPLE 10 - Low Rosin Water-Free Alcoholic Flux

(a) TAM Formulation no. 3 (BASF) of EXAMPLE 6 (a) was used directly with commercially-obtained water-white rosin, by dissolving 10.98 g TAM in 65 ml

IPA and 56.78 g water-white rosin in about 200 ml IPA, combining these two solutions and adding additional IPA to a final water-free alcoholic solution volume of 3785 ml or 1 U.S. gal.

5 The flux, by calculation, contained 56.78 g rosin (1.9%), 10.98 g TAM (0.4%) and 2932.02 g IPA (97.7%), total 2999.78 g of sp.g. 0.7925 (2999.78/3785), at a rosin content of 1.9% corresponding to 1.5 vol.% (56.78/3785).

10 (b) The procedure of (a) was repeated using instead TAM Formulation no. 4 (Rhone-Poulenc) of EXAMPLE 6 (c).

15 (c) The procedure of (a) was repeated using instead TAM Formulation no. 1 (DuPont Canada) of EXAMPLE 1.

Table I shows the proportions (gms, % and parts) of the ingredients in the fluxes of Examples 9-10.

TABLE I - Low Rosin Water-Free Fluxes of the Invention

	Ex. 9		Ex. 10	
	Grams	Wt. %	Grams	Wt. %
Rosin	198.02	6.1	56.78	1.9
TAM	15.00	0.5	10.98	0.4
IPA	3052.76	93.4	2932.02	97.7
Flux	3265.78	100	2999.78	100
Rosin	198.02	93.0	56.78	83.8
TAM	15.00	7.0	10.98	16.2
Active	213.02	100	67.76	100
Active	213.02	6.5	67.76	2.3
Other	3052.76	93.5	2932.02	97.7
Flux	3265.78	100	2999.78	100
Rosin	198.02	pts.	56.78	pts.
TAM	15.00	8	10.98	19
IPA	3052.76	1542	2932.02	5164
Sp.G.	0.8628	0.7925		
Volume	3875 ml	3875 ml		
Volume	1 U.S. gal.	1 U.S. gal.		

Table I shows that the fluxes of EXAMPLES 9-10 have 2-6% rosin, 0.4-0.5% TAM and 93-98% IPA, or 2-7% active and 93-98% other components. At 100 pts rosin, they have 8-19 pts TAM and 1542-5164 pts IPA.

5 The flux of EXAMPLE 9 is used by foam or wave application, and that of EXAMPLE 10 by spray application, in soldering circuit board components, typified in EXAMPLE 11. When used by spray application, the rosin content is more dilute, e.g.,
10 at most, 1-2% rosin (1-2g/100ml), to form a fine mist.

EXAMPLE 11

Each of the three TAM-based fluxes (BASF, Rhone-Poulenc and DuPont Canada) of each of EXAMPLES
15 6-10 was individually used on an electronic circuit board with pre-tin-lead-plated through holes and components, by foam or wave application (fluxes of EXAMPLES 6-9) or spray application (fluxes of EXAMPLE 10), soldered at 250-260°C with a solder of a eutectic
20 tin-lead 63% Sn - 37% Pb composition (mp 183°C), or of a tin-lead 60% Sn - 40% Pb composition (mp 191-193°C). In each case, the three-acid mixture did not sublime and condense on the board, and a hotter-than-usual ambient condition was not needed to avoid this
25 problem.

Each of the fluxes of EXAMPLES 6-10 showed a significant improvement in solderability, providing an increased production yield per a reduced number of voids and shorts, compared to the currently used known
30 flux no. 2 (LONCO RF 710, an activated 5% rosin flux of undisclosed proprietary formulation, London

Chemical Co., Bensenville, IL). The surface insulating property was deemed excellent, allowing the soldered circuit boards to be conformal-coated (i.e., to have a conventional protective coating applied thereon) directly, without washing to remove ionic residue, and prior to the usual reliability tests, whereas normally conformal-coating is only performed after the boards have been cleaned with Freon TMS and have passed given reliability tests.

These beneficial results were unexpected, even though the 5% rosin content known flux no. 2 has a higher rosin content than the roughly 3% rosin content EXAMPLE 6-7 fluxes, because the same beneficial results also occurred with the 5% rosin content EXAMPLE 8 fluxes and the 6.1% rosin content EXAMPLE 9 fluxes, confirming that the problem is not caused by the rosin content, but by the high ionic residue left at the soldering site, which is traceable to the high ionic content in the known flux.

The presence of water is not critical as it boils off during soldering, and may be added to reduce solvent, e.g., IPA, and cost and to aid foaming without adding a foaming agent, in up to a minor amount of the total liquid in the high rosin content fluxes of EXAMPLES 1-4, as noted in Table G, for general metal soldering (EXAMPLE 5) where splattering is not a major problem. In the low rosin content fluxes of EXAMPLES 6-10 for circuit board soldering (EXAMPLE 11), water is present in only a slight amount, as noted in Tables H and I, to avoid this problem.

While a higher TAM content is used to activate the higher rosin content fluxes for stronger cleaning in general metal soldering, a lower TAM content is used in the lower rosin content fluxes for milder cleaning in circuit board soldering.

Production Line Test Run

Circuit boards designated Parts no. 1-4 were soldered per EXAMPLE 11 with 3% rosin content flux of EXAMPLE 7 (c) [TAM Formulation no. 1 (DuPont Canada) of EXAMPLE 1] on a wave-soldering production line in a three-day test run, with a comparison run of Parts no. 1-4 soldered with known flux no. 2 (LONCO RF 710).

Table J shows pertinent specific gravity data of the run:

TABLE J - Specific Gravity of Invention Flux
Three-Day Test Run - 3% Rosin Flux
Wave-Soldering Line - Entire Day Shift

<u>Control Point</u>		<u>Reading</u>	<u>Time</u>
Day 1 - Sp.G. Set Point	Sp.G. Reading	0.810	0:00 hr - Start of Test Run
		0.815	8:30 hr - End of Test Run
Day 2 - Sp.G. Set Point	Sp.G. Reading	0.810	0:00 hr - Start of Test Run
		0.816	8:50 hr - End of Test Run
Day 3 - Sp.G. Set Point	Sp.G. Reading	0.810	0:00 hr - Start of Test Run
		0.811	9:40 hr - End of Test Run

Table K shows pertinent flux and make-up
isopropyl alcohol usage and rejection rate comparison
data of the run.

TABLE K - Usage of Invention Flux and Reject Rate Comparison
Three-Day Test Run - 3% Rosin Flux
Wave-Soldering Line - Entire Day Shift

Part	U.S.Gals.	Start U.S.Gals.	Flux Added U.S.Gals.	Usage IPA Added U.S.Gals.	Reject Rate Comparison (Averaged ppm)		
					Invention Flux ppm (%)	Known Flux #2 ppm (%)	
Day 1 - #1	2.75	0	0	1	0 (None)	1000 (0.10)	
#2	-	0	0	1	1500 (0.15)	2375 (0.24)	
#3	-	0	0	1	1000 (0.10)	2250 (0.23)	
Day 2 - #1	-	1	1	3	666 (0.07)	1000 (0.10)	
Day 3 - #2	2.75	0	0	2*	500 (0.05)	2375 (0.23)	
#4	-	0	0	2	Not Available		
Totals	5.5	1	1	10	3666 (0.37)	9000 (0.90)	
Mean					733 (0.07)	1800 (0.18)	

* High humidity - hot day, higher evaporation rate.

The data in Tables J and K typify consumption of the 3% rosin content flux of the invention under wave-solder production line conditions over a three-day period of 8:30 to 9:40 hour/day plant shifts, formed of two consecutive days (Days 1 and 2) and a third day (Day 3) after a temporary, unrelated, interruption of three intervening days.

A titration test was performed at the end of each day to determine the rosin content of the flux in the wave-soldering machine used, and it was indicated to be at the required level, consistent with the Table J specific gravity readings.

Table K shows that the total flux usage was 6.5 U.S.gals. plus 10 U.S. gals. isopropyl alcohol added to make up evaporation losses. The line was serviced with 2.75 U.S. gal. amounts twice for two separate start-ups (Days 1 and 3), and these lasted for the entire shift without requiring further flux, while only one addition of 1 U.S. gal. of flux was required on the intermediate day (Day 2) to that remaining from the previous day (Day 1).

Isopropyl alcohol was added periodically to the flux to maintain the required low rosin concentration. A small amount of the flux thickened when left uncovered for more than 24 hours, but was made fully usable again by re-mixing or adding make-up amounts of isopropyl alcohol. Also, high humidity weather conditions caused loss of isopropyl alcohol from the flux at a higher than normal evaporation rate.

As to odour, the flux of the invention had a pleasant smell as noticed when pouring it into

the wave-soldering machine tank, and when it came into contact with the molten solder during soldering, there was no vulgar or offensive smell as occurred with known flux no. 2 in the comparison runs. As to solderability, visual inspection of boards indicated an enhanced cosmetic appearance to the solder joints per the flux of the invention that did not occur with known flux no.2. The flux of the invention improved soldering characteristics as shown by reduced bridging and voids compared to known flux no.2. This improvement in characteristics was coupled with a reduction in the amount of touch-up required on the boards compared to that with known flux no. 2.

The flux of the invention led to a higher soldered product yield per shift than with known flux no.2, as shown by the parts per million (ppm) rejection rate data in Table K. No Parts no.1 soldered with the invention flux were rejected on Day 1 compared to a 0.10% reject rate for known flux no.2, whilst the rate for Parts no. 2 was 0.15% for the flux of the invention compared to 0.24% for known flux no. 2, and for Parts no. 3 was 0.10% compared to 0.23% for known flux no.2. On Day 2, for Parts no.1 it was 0.07% for the flux of the invention compared to 0.10% for known flux no. 2. On Day 3, for Parts no. 2 it was 0.05% for the flux of the invention compared to 0.23% for known flux no.2, and whilst the Parts no.4 rates are not available, they presumably follow the other data.

Statistically, the five separate ppm rejection rate values for the flux of the invention total 3666 ppm or 0.37%, averaging 733 ppm or 0.07%, whilst those for known flux no. 2 total 9000 ppm or 0.90%, averaging 1800 ppm or 0.18%. Thus, the

invention flux resulted in a reduction of the rejection rate by much more than half that of the known flux, and in one instance (Day 1, Part no. 1) the flux of the invention unexpectedly afforded a production line product at 100% acceptability or at zero rejection rate.

Product Reliability Tests

Twenty completed printed circuit board assemblies or mainboards, i.e., attached to keyboard and display driver board components of the intended final unit, produced during the three-day test run using the 3% rosin content flux of the invention were subjected to product reliability tests. These twenty mainboards were not washed with cleaning liquid to remove soldering flux residue before being conformal-coated.

All such mainboards were directly conformal-coated by a conventional step in production line soldering of the boards to protect their electrical components from ambient moisture and the like. Normally, washing is required before conformal-coating to remove flux residue to minimize defects from developing in use of the boards, even though they have a moisture-protective conformal-coating as an added precaution.

Fifteen of these mainboard units were subjected to the following conventional tests:

(1) 500 hours Power-Temperature Cycle, with an applied voltage of 14 volts, at -40 to +85°C, and 5 minutes cycle time, and then

(2) 168 hours Bias High Voltage Humidity, at 11 volts bias, 65°C and 95% relative humidity (rel. hum.)

Nine of these fifteen mainboard units were thereafter subjected to the following conventional test, along with the remaining five untested mainboard units as controls:

- 5 (3) Moisture Susceptibility, at -40°C for 2 hours, and then at 65°C and 95% rel. hum. for 2 minutes, after which the unit is powered for 5 minutes to verify that it is operational.

- 10 All the mainboards performed satisfactorily in these reliability tests, and it was determined that the solderability of the flux of the invention was excellent. There was a significant improvement in solderability and increase in yield per a reduction in the number of voids and shorts, compared to known flux no. 2. The surface-insulating property of the flux of the invention was found to be excellent, allowing the boards to be directly conformal-coated without washing and before being subjected to the reliability tests that usually precede conformal-coating.
- 15
- 20

Total Ionics and Extracted Anions Cleanliness Tests

- Three circuit boards (boards 1-3), soldered using the 3% rosin flux [TAM Formulation no.1 (DuPont Canada)], were subjected to successive (a) and (b) cleanliness tests to determine ionic residue levels:
- 25

- (a) first with total ionics measurement being performed using an Ionic Contamination Test System Omega (RTM) Meter (Kenco Industries Inc.), and
- 30 (b) thereafter with total extracted anions measurement, i.e., including weak organic acid residue plus background anion residue of extraneous ions such as bromine and chlorine ions from the board

itself, being performed using an ion chromatograph, Dionex (RTM) Ion Chromatograph Model 4000I CMA-3 (Dionex Co.),

5 in each case without washing and before conformal-coating.

Five additional circuit boards (boards 4-8), soldered in the same manner using that 3% rosin flux, were subjected to these tests before applying the conformal-coating but, in this case, whilst the tests were performed on the first two boards (boards 4-5) without washing, the last three (boards 6-8) were washed with Freon (RTM) TMS to remove ionic residue.

Four further circuit boards (boards 9-12), soldered in the same manner using known 5% rosin flux, i.e., known flux no. 2 (LONCO RF 710) for three boards (boards 9-11) and a further known flux no. 3 (HI-GRADE 3565D, an activated 5% rosin flux of undisclosed proprietary formulation, High-Grade Alloy Corp., East Hazelcrest, IL) for the fourth board (board 12), were also tested before conformal-coating, with the tests being performed on the first two boards (boards 9-10) without washing and on the second two (boards 11-12) after washing with Freon (RTM) TMS.

Moreover, five additional boards (boards 13-17), soldered in the same manner using known flux no.2, and five additional boards (boards 18-22), soldered in the same manner using known flux no.3, were also tested after washing with Freon (RTM) TMS but before conformal-coating, and their averaged total extracted anionic residue values, respectively, were calculated.

All boards (boards 1-22) were of the same size, having a total area of 230 cm^2 (total of both sides of the board), such that the $\mu\text{g}/\text{cm}^2$ values are based on that 230 cm^2 board area.

5 The (a) Omega (RTM) Meter tests were carried out by immersing the entire board in a solvent mixture of 75% isopropyl alcohol (IPA) and 25% de-ionized water at room temperature, and
10 subjecting the board to continuous measurement for about 6 minutes at room temperature, according to a common standard procedure.

 The (b) ion chromatograph tests for total anions were carried out according to a special extraction procedure in which the board was extracted
15 with 200 ml of a solvent mixture of 75% IPA and 25% de-ionized water for 1 hour at 80°C , to form an extract concentrate of attendant anions in about 40 ml residual IPA-water mixture (mainly water as the 80°C temperature promotes IPA volatilization).
20 The extraction causes uptake into the concentrate of weak organic acid from the flux residue, plus mainly bromine and chlorine ions from constituents of the board, for measurement of total extracted anions.

 This procedure is performed by placing the
25 board in a commercially obtained Kapak/Scotch Pak heat-sealable pouch (Kapak Corp., Minneapolis, MN), and sealing the pouch for the extraction procedure, so that during extraction the volatiles boiling off at the 80°C ambient temperature are evacuated from
30 the sealed pouch. The extract concentrate is subjected to standard chemical analysis of all anions, including those of organic acids traceable to the flux, in the ion chromatograph.

Although the (a) Omega (RTM) Meter tests measure total ionics traceable to the flux constituents by a rapid (6 minute) test procedure, whilst the (b) ion chromatograph tests measure total anions from all attendant sources by a prolonged (1 hour) intensive extraction procedure, the levels of ionic residue in both measurement procedures (a) and (b) are in the same units of $\mu\text{g}/\text{cm}^2$. However, the (a) Omega (RTM) Meter units are in terms of the NaCl equivalent thereof, as the Omega (RTM) Meter was calibrated with a standard known concentration of NaCl, while the (b) ion chromatograph units are direct measures of extracted anionic content per ion chromatograph measurement.

Table L shows the unexpected results obtained in these cleanliness tests, for all of these 230cm^2 area boards, (a) with the total ionic residue expressed in micrograms per square centimetre, as sodium chloride equivalent (Omega (RTM) Meter), and (b) with the total extracted anionic residue expressed in micrograms per square centimetre (ion chromatograph):

TABLE L - Cleanliness Testing

Board	(a)		(b)		Condition
	Omega Meter Total Ionics Ionic ₂ Residue µg/cm ² - As NaCl Equivalent	Chromatograph Total Extracted Anions Anionic Residue µg/cm ²	Ion		
Invention					
1	2.446	21.558*			Unwashed
2	2.668	21.558*			Unwashed
3	2.752	21.558			Unwashed
4	4.116	39.172			Unwashed
5	3.727	33.753			Unwashed
6	2.231	30.434			Washed
7	2.011	20.669			Washed
8	2.512	20.214			Washed
Flux 2					
9	4.623	105.263			Unwashed
10	4.513	111.513			Unwashed
11	8.101	68.281			Washed
Flux 3					
12	8.413	24.637			Washed
Flux 2					
13-17	-	40.63**			Washed
Flux 3					
18-22	-	52.16**			Washed

* Boards 1-3: 64.674 µg/cm² total anions; 21.558 µg/cm² average per board.

** Averaged value for five boards.

The (a) Omega (RTM) Meter total ionics results show that washing the boards to remove ionic residue traceable to the flux constituents is superfluous for the flux of the invention as very low ionic residues, all be low a 5 $\mu\text{g}/\text{cm}^2$ (NaCl equivalent) trace amount, and mainly below a 3 $\mu\text{g}/\text{cm}^2$, are obtained without washing (boards 1-5), and that washing only slightly further decreases the ionic residue (boards 6-8).

On the other hand, per the (a) tests, washing the circuit board (board 11) soldered with the known flux no. 2 appears to increase the ionic residue level compared to the unwashed boards (boards 9-10), and the similar high level of the washed board (board 12) soldered with the known flux no. 3 is consistent with this conclusion. This increase in ionic residue level on washing must be due to the undisclosed high ionic content in the fluxes no. 2 and no. 3, since rosin is not ionic and cannot alone explain this increase in ionic level on washing (cf. the explained results obtained in EXAMPLES 5 and 11).

The (b) ion chromatograph test results show similar differences among the boards, the values being higher than those of the (a) tests, as the (b) tests measure total anion levels from all attendant sources, including the board itself, and are based on a vigorous extraction of anionic constituents that would not necessarily cause corrosion in the unextracted state.

As to the boards soldered with the flux of the invention, per the (b) tests, the total extracted anionics levels of the washed boards (boards 6-8), 20-30 $\mu\text{g}/\text{cm}^2$, compared to the unwashed boards (boards 1-5), 22-39 $\mu\text{g}/\text{cm}^2$, confirm that washing the boards is

superfluous for the flux of the invention.

As to the boards soldered with the known fluxes no. 2 and no. 3, per the (b) tests, the lower total extracted anionics levels of the twelve washed
5 boards (boards 11-22), 25-68 $\mu\text{g}/\text{cm}^2$, compared to the higher levels of the two unwashed boards (boards 9-10), 105-112 $\mu\text{g}/\text{cm}^2$, is no doubt due to the vigorous extraction used for the (b) tests. These (b) tests may be regarded as mimicking more pronouncedly the
10 extractive action per the (a) tests of the Freon (RTM) TMS washing liquid in reaching ionic constituents in any attendant rosin residue serving as retaining matrix for such ionic constituents of the highly acidic known fluxes no. 2 and no. 3 on the washed
15 boards (boards 11-12), compared to the unwashed boards (boards 9-10).

Thus, a plausible explanation of the role of the rosin is that any content thereof in the flux residue remaining after soldering acts as a matrix
20 which retains ionic constituents in direct proportion to the initial concentration of such constituents in the flux, and in direct relation to the capability (i.e., relative ease or difficulty) or incapability of such ionic constituents to volatilize during the
25 heat-soldering operation. This matrix retention explanation as to the differences in results in using the invention flux compared to known fluxes no. 2 and no. 3, as shown in Table L, is consistent with the differences in results in using the flux of the
30 invention compared to known fluxes no. 1 and no. 2 per
EXAMPLES 5 and 11.

In any case, per the (b) tests, the total extracted anionics levels of the unwashed boards soldered with the flux of the invention (boards 1-5) are markedly below the levels of the unwashed boards soldered with the known flux no. 2 (boards 9-10), and the total extracted anionics levels of the washed boards soldered with the flux of the invention (boards 6-8) are markedly below the levels of the washed boards soldered with the known fluxes no.2 and no.3 (boards 11 and 13-22), with one exception (board 6 compared to board 12). Nevertheless, as to all (b) test values, those of the unwashed boards soldered with the flux of the invention (boards 1-5) are sufficiently low in anionic residue to obviate the need for a washing step.

Accordingly, it can be appreciated that the specific embodiments described are merely illustrative of the general principles of the invention.



Claims:

1. A low-residue soldering flux comprising rosin and an activating amount of an essentially non-subliming dibasic acid mixture containing by weight
5 15-30% succinic acid, 40-63% glutaric acid and 10-30% adipic acid, said dibasic acid mixture having a boiling point of 150-210°C.
2. A low-residue soldering flux according to claim 1, in which the relative proportion of the
10 rosin and dibasic acid mixture to each other by weight in the flux is in the range of 65% rosin - 35% dibasic acid mixture to 95% rosin - 5% dibasic acid mixture.
3. A low-residue soldering flux according to claim 1, in which the flux includes a volatilizable
15 flux vehicle.
4. A low-residue soldering flux according to claim 3, in which the rosin and dibasic acid mixture are present in a combined amount of 1.25-55% by weight and the vehicle is present in an amount of
20 45-98.75% by weight.
5. A low-residue soldering flux according to claim 4, in which the relative proportion of the rosin and dibasic acid mixture to each other by weight in the flux is in the range of 65% rosin - 35% dibasic
25 acid mixture to 95% rosin - 5% dibasic acid mixture.
6. A low-residue soldering flux according to claim 3, in which the vehicle comprises 70-100% by weight of a volatile organic solvent and 0-30% by weight of de-ionized water.



7. A low-residue soldering flux according to claim 6, in which the solvent is selected from an aliphatic or cycloaliphatic alcohol, an aliphatic carboxylic acid ester, a cyclic ketone, or mixtures thereof.

8. A low-residue soldering flux according to claim 7, in which the solvent is a lower aliphatic alcohol having a chain of less than 5 carbon atoms.

9. A low-residue soldering flux according to claim 7, in which the solvent and water are in the relative proportion to each other by weight of 70-99.5% solvent and 0.5-30% de-ionized water.

10. A low-residue soldering flux according to claim 9, in which the solvent is isopropyl alcohol.

11. A low-residue soldering flux consisting essentially of, by weight, 1-35% rosin; 0.25-20% of an essentially non-subliming dibasic acid mixture containing 15-30% succinic acid, 40-63% glutaric acid and 10-30% adipic acid; 45-98.75% of a volatilizable organic solvent; and 0-20% de-ionized water; said percentages of rosin, dibasic acid mixture, solvent and water being based on the weight of the total flux, and said percentages of succinic, glutaric and adipic acids being based on the total weight of the dibasic acid mixture, the dibasic acid mixture having a boiling point of 150-210°C.

12. A low-residue soldering flux according to claim 11, in which the flux contain 25-35% rosin, 2.5-20% dibasic acid mixture, 45-60% solvent and 5-20% de-ionized water.



13. A low-residue soldering flux according to claim 12, in which the solvent is isopropyl alcohol.

5 14. A low-residue soldering flux according to claim 11, in which the flux contains 1-8% rosin, 0.25-1% dibasic acid mixture, 90-98.75% solvent and 0-1% de-ionized water.

10 15. A low-residue soldering flux according to claim 14, in which the solvent is isopropyl alcohol.

15 16. A method of heat-soldering metal surfaces which comprises applying a solder to the metal surfaces in the presence of an effective amount of a low-residue flux according to claim 1 in coating disposition on the metal surfaces, and at a temperature sufficient for molten flow of the solder and for volatilization of the flux from the metal surfaces, so as to leave the surfaces essentially free from ionic residue from the flux.

20 17. A method of heat-soldering metal surfaces according to claim 16, in which the soldering is effected at a temperature of 200-500°C.

25 18. A method of heat-soldering metal surfaces according to claim 16, in which the soldering is effected with a tin-lead solder.

30 19. A method of heat-soldering metal surfaces according to claim 16, in which the flux includes a volatilizable flux vehicle, and soldering is carried out at a temperature sufficient to leave



at most a trace amount of ionic residue on said metal surfaces.

20. A method of heat-soldering metal surfaces which comprises applying a solder to the metal surfaces in the presence of an effective amount of a low-residue flux according to claim 12 in coating disposition on the metal surfaces, and at a temperature sufficient for molten flow of the solder and for volatilization of the flux from the metal surfaces, so as to leave the metal surfaces essentially free from ionic residue from the flux.

21. A method of heat-soldering metal surfaces comprising applying a solder to the metal surfaces in the presence of an effective amount of the flux of claim 14 in coating disposition on the metal surfaces, and at a temperature sufficient for molten flow of the solder and for volatilization of the flux from the metal surfaces, so as to leave an ionic residue on said metal surfaces of at most about $5 \mu\text{g}/\text{cm}^2$ equivalent of sodium chloride.

22. A method of heat-soldering metal surfaces according to claim 21, in which the heat-soldering leaves an ionic residue of at most about $3 \mu\text{g}/\text{cm}^2$ equivalent of sodium chloride on said metal surfaces.

23. A soldered product produced by a method according to claim 21.

24. A soldered product produced by a method according to claim 22.

25. A low-residue, high-rosin content aqueous alcoholic flux substantially as hereinbefore particularly described in any one of Examples 1 to 4.

26. A low-residue, low-rosin content



aqueous alcoholic flux substantially as hereinbefore particularly described in any one of Examples 6 to 8.

27. A low-residue, low-rosin content, water-free alcoholic flux substantially as hereinbefore particularly described in any one of Examples 9 to 10.
