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[54] **FLUX FOR USE IN SOLDERING OF STAINLESS STEELS**

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[51] **Int. Cl.** **C09d 1/00**

[58] **Field of Search** 148/26, 6.15, 24; 206/286; 252/79.2; 106/286; 29/471.1, 496

[56] **References Cited**

UNITED STATES PATENTS

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2,987,817 5/1931 Koziik 148/26

FOREIGN PATENTS OR APPLICATIONS

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[57] **ABSTRACT**

Fluxes for use in soldering stainless steels comprising compositions containing a major proportion of orthophosphoric acid and a minor proportion of a mono- or diammonium phosphate, or an amine mono- or diorthophosphate, advantageously in the presence of water, and desirably in admixture with various supplemental materials.

12 Claims, No Drawings

FLUX FOR USE IN SOLDERING OF STAINLESS STEELS

This invention relates to novel fluxes for use in soldering of stainless steels and to the soldering of stainless steels with said fluxes.

In the soldering of stainless steels, various fluxes have been suggested, such as zinc chloride or zinc chloride and hydrochloric acid, but they are corrosive, commonly cause pitting, and have been found to be quite unsatisfactory. Where they are utilized, it is necessary to neutralize the flux residues and then to wash with water. Apart from the time consumption involved in such operations, it is, at times, difficult to get access to such residues, and pitting and corrosion commonly cannot be avoided. Perhaps the most satisfactory of the fluxes used in the soldering of stainless steels is orthophosphoric acid. However, orthophosphoric acid, too, has certain objections. While it is generally non-corrosive towards stainless steels during the actual fluxing operations, it fails to bring about fully satisfactory soldered joints due to the fact that it has inadequate wetting, spreading and capillary action, even when used in high concentrations.

It has been found, in accordance with the present invention, that markedly improved fluxes for use in soldering stainless steels can be made by incorporating into a phosphorus-containing acid minor proportions of one or more mono- and diammonium phosphate salts and/or one or more amine mono- and disalts of phosphoric acids, hereafter described in detail, and, in the particularly preferred embodiments of the invention, certain additional ingredients. The resulting fluxes are characterized by excellent resistance to corrosive effects, and substantially improved wetting, spreading and capillary action. The result is that they enable clean, soldered joints to be obtained which are characterized by excellent strength due to good surface coverage of the interface between the stainless steel and the metal to which it is soldered which, desirably, is also stainless steel.

The major ingredient of the improved fluxes of the present invention, as indicated above, is a phosphorus-containing acid. Orthophosphoric acid is most advantageously utilized, both from the standpoint of its functioning and its commercial availability and low cost, but phosphorous acid (usually available in aqueous solution in 70–72 percent concentrations) can be employed. The orthophosphoric acid may be in the form of aqueous orthophosphoric acid containing as low as 40 percent orthophosphoric acid, or it may be as high as about so-called commercial 115 percent phosphoric acid, but it is preferred to utilize about 75 to 105 percent orthophosphoric acid.

There is admixed with the orthophosphoric acid or other phosphorus-containing acid one or more of monoammonium phosphate ($NH_4H_2PO_4$) or diammonium phosphate $(NH_4)_2HPO_4$ or monobasic or dibasic organic amine phosphates, the organic amine containing from one to six carbon atoms.

Illustrative of the foregoing amines are amines selected from the group consisting of alkylamines (including cycloalkylamines), alkenylamines, hydroxyalkyl mono- and diamines, alkylenepolyamines, and heterocyclic amines, and they may be saturated or unsaturated, straight chain or branched chain, although it is particularly preferred that they be saturated, straight chain alkylamines. Said amines should be unsubstituted and they will, therefore, contain only carbon, hydrogen and nitrogen, except in the case of those amines of the types referred to above which contain hydroxy groups and those which may contain oxygen in an ether grouping.

Illustrative examples of the foregoing types of amines, which examples may be selected from a wide group, include such alkylamines (including cycloalkylamines) as methylamine, dimethylamine, ethylamine, diethylamine, triethylamine, n-propylamine, isopropylamine, n-butylamine, isobutylamine, n-amylamine, isoamylamine, n-hexylamine, isohexylamine, and cyclohexylamine; alkenylamines such as allylamine and methallylamine; alkylenepolyamines such as ethylenediamine, diethylenetriamine and triethylenetetramine; hydroxyalkyl mono- and diamines such as monoethanolamine, diethanolamine, triethanolamine, and commercial mixtures of said ethanolamines, n-propanolamine, monoisopropanolamine, diisopropanolamine, and commercial mixtures of said isopropanolamines, butanolamines, aminoethyl ethanolamine, ethyldiethanolamine, diethylethanolamine, 2-amino-2-methyl-1propanol, and 2-amino-1-butanol, and glycerolamines; hydroxyalkyl alkylene polyamines such as hydroxyethyl ethylenediamine and hydroxyethyl diethylenetriamine; polyoxyalkyleneamines such as are represented by the formula

$$H_2N—C_2H_4—(O—C_2H_4)_n—NH_2$$

where n is an integer from 1 to 2; aniline; and heteroxyclic amines such as morpholine, pyridine, methylpyridine, piperidine and methylpiperidine; and compatible mixtures of any two or more thereof.

The foregoing ammonium and organic amine phosphates may be preformed and admixed, as such, with the phosphorus-containing acid, notably orthophosphoric acid, or they may be formed in situ by adding ammonia or ammonium hydroxide, or the organic amine, to the phosphorus-containing acid in requisite or controlled amounts. The said ammonium and/or organic amine phosphates may comprise from as low as about 10 percent to as high as about 50 percent, by weight, of the phosphorus-containing acid employed, for instance, 75 percent orthophosphoric acid. While still greater quantities can be used, it is unnecessary to do so. In most cases at least, the proportions of said phosphate salts will range from about 15 to about 35 percent by weight of the phosphorus-containing acid. It may also here be pointed out that it is preferred that the soldering flux be in the form of a liquid, although it can be converted into a paste, if desired. Furthermore, when used in the form of a liquid, it is desirable that the ammonium or organic amine phosphate salts be in solution in the phosphorus-containing acid. However, the fluxes are operative even in those instances where said phosphate salts may be used in amounts as to exceed their solubility in the flux composition.

It is generally advantageous to incorporate added water as an ingredient of the flux compositions of the present invention. To be sure, water is present in the phosphorus-containing acids of commerce, the amount thereof varying depending, for instance, on whether said acid is 75, 105 or 115 orthophosphoric acid. The added water serves, among other things, to provide for a flux of liquid character, of desired viscosity, aids in effecting solution of the ammonium and/or organic amine phosphate salts in the flux compositions, and brings about improved fluxing action. Generally, it is desirable to add about 10 to about 15 percent water, based on the weight of the phosphorus-containing acid, for instance, 75–105percent orthophosphoric acid.

Various supplemental agents can be added to obtain particular effects. Thus, for instance, surfactants, especially of the nonionic type and exemplified by normally solid, paste or liquid "PLURONICS" (Wyandotte Chemicals Corp.) and "IGEPAL CO–730" (GAF Corporation) are desirably incorporated. The "PLURONICS," as is well known, are condensates or adducts of ethylene oxide with hydrophobic bases, in the form of polyoxypropylene glycols generally having a molecular weight of 1200 or higher, and are disclosed, for example, in U.S. Pat. Nos. 2,674,619 and 2,677,700. Other nonionic surfactants can be used such as ethylene oxide adducts of C_{12}–C_{20} linear and branched chain alcohols, including Oxo alcohols, and ethylene oxide adducts of C_9–C_{18} alkyl phenols, said nonionic surfactants being, per se, well known and being disclosed in many U.S. patents as, for instance, in U.S. Pat. Nos. 1,970,578; 2,965,678 and as intermediates in No. 3,004,056. "IGEPAL CO–730" is a nonylphenoxypoly (ethyleneoxy) ethanol and is exemplary of said latter surfactants. The nonionic surfactants function, in certain instances, to improve the homogeneity of the fluxes, and they also tend to enhance the wetting and spreading properties of the fluxes during soldering operation. The surfactants, where utilized, are desirably employed in amounts of about 0.1 to 1 percent, particularly 0.2 to 0.5 percent by weight of the phosphorus-containing acid.

The following examples are illustrative of the preparation of the improved fluxes made in accordance with the invention. It will be understood that numerous other fluxes can be made in the light of the guiding principles and teachings disclosed above. All parts listed are by weight.

EXAMPLE 1

Orthophosphoric acid (75%)	100
Monoammonium phosphate	36
Water	14
"IGEPAL CO–730"	0.4

The ingredients may be mixed together in any suitable order. Thus, for instance, the monoammonium phosphate may be dissolved in a water solution of the "IGEPAL CO–730," and then the orthophosphoric acid is added, with stirring. The resulting flux is a liquid of slightly viscous character.

EXAMPLE 2

Orthophosphoric acid (75%)	100
Di-ammonium phosphate	17
Water	7
"IGEPAL CO–730"	0.3

EXAMPLE 3

Orthophosphoric acid (57%)	100
Monoammonium phosphate	33
Water	15

EXAMPLE 4

Orthophosphoric acid (75%)	100
Monoammonium phosphate	23
Di-ammonium phosphate	6.5
Water	15

EXAMPLE 5

Orthophosphoric acid (105%)	100
Ammonium hydroxide (28%) (to be added gradually to the phosphoric acid)	19
Water	28

EXAMPLE 6

Orthophosphoric acid (75 %)	100
Ethylamine orthophosphate (diabasic)	16
Water	15
"IGEPAL CO–730"	0.3

EXAMPLE 7

Orthophosphoric acid (75%)	100
Isopropylamine orthophosphate (monobasic)	26
Water	13

EXAMPLE 8

Orthophosphoric acid (75%)	100
Cyclohexylamine orthophosphate (dibasic)	25
Water	14
"IGEPAL CO–730"	0.4

EXAMPLE 9

Orthophosphoric acid (75%)	100
Isobutylamine orthophosphate (dibasic)	22
Water	13

EXAMPLE 10

Orthophosphoric acid (75%)	100
Monoammonium orthophosphate	22
Ethylamine orthophosphate (dibasic)	8
Water	13
"IGEPAL CO–730"	0.4

EXAMPLE 11

Phosphorous acid (70–72%)	100
Monoammonium orthophosphate	26
Water	13
"IGEPAL CO–730"	0.4

EXAMPLE 12

Orthophosphoric acid (75%)	100
Aniline orthophosphate (monobasic)	20
Water	12

The stainless steels which can be soldered with the improved fluxes of the present invention can be chosen from among the many which are well known to the art. Illustrative of such stainless steels are those containing chromium, for instance, of the order of 18 percent chromium; those containing chromium and titanium, for instance, those containing of the order of 12 percent chromium and 2 percent titanium; those containing varying proportions of chromium and nickel; and those containing varying proportions of chromium, titanium, nickel and vanadium. Illustrative of such stainless steels are those sold commercially under the trade designations 300 series and 400 series, and "TI-CHROME" (Crucible Steel Corporation). The invention is of especial value in connection with the soldering of stainless steel sheets to form gutters and the like using, for instance, series 304 stainless steels.

In soldering the stainless steels to form seams or joints with various metals, which latter may be stainless steels of the same or different character, or which may be copper, copper-base alloys and copper-containing alloys, or non-stainless steels, and the like, various solders can be employed such as, for instance, 50 percent tin-50 percent lead; 60 percent tin-40 percent lead; 95 percent tin-5 percent antimony; 40 percent tin-60 percent lead; 30 percent tin-70 percent lead; and variants of such solders and other known soft solders. It is especially desirable to use approximately 50 percent tin-50 percent lead solders since they have good flow properties at relatively low temperatures. Conventional soldering techniques and conventional soldering temperatures are used with the improved fluxes of the present invention, being employed in the same general manner in which zinc chloride, or zinc chloride in admixture with hydrochloric acid, or orthophosphoric acid have heretofore been used as a flux so that no detailed explanation is necessary. Where gutters are made, for instance, the heat utilized in the soldering operation may be supplied by a hot iron or a torch or the like.

What is claimed is:

1. A flux for use in soldering stainless steels consisting essentially of, a major proportion of at least one phosphorus-containing acid selected from the group of phosphoric acid and phosphorous acid, and from about 10–15 percent, by weight of said phosphorus-containing acid of at least one phosphate salt selected from the group of monoammonium phosphate, diammonium phosphate, and amine mono- and diphosphates in which the amine contains from one to six carbon atoms.

2. The flux of claim 1 in which said flux is in the form of a liquid containing added water.

3. The flux of claim 2 in which the phosphate salt is at least one member of the group of monoammonium phosphate and diammonium phosphate in solution in said liquid.

4. The flux of claim 1 in which the phosphorus-containing acid comprises 75–105 percent orthophosphoric acid.

5. The flux of claim 4 in the form of a liquid.

6. The flux of claim 4 which includes a minor proportion of a nonionic surfactant.

7. The flux of claim 5 which includes a minor proportion of a nonionic surfactant.

8. A flux in accordance with claim 5 wherein the following ingredients are present in approximately the stated parts by weight:

Orthophosphoric acid (75-105%)	100
Monammonium and/or diammonium phosphate	11 15–35
Water	10–15

9. A flux in accordance with claim 8 wherein the following ingredients are present in approximately the stated parts by weight:

Orthophosphoric acid (75%)	100
Monoammonium phosphate	36
Water	14

10. A flux in accordance with claim 8 wherein the following ingredients are present in approximately the stated parts by weight:

Orthophosphoric acid (75%)	100
Diammonium phosphate	17
Water	7

11. The flux of claim 2 in which the amine is an alkylamine containing from one to three carbon atoms.

12. The flux of claim 11 in which the phosphorus-containing acid is 75-105 percent orthophosphoric acid.

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