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WORKING OF METALS WITH SALTS OF AMINE CARBOXYLIC ACIDS AND AMINE ALKYL PHOSPHATE

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This invention relates to the working of metals and more particularly to the use of a novel drawing compound therein.

Reference to the working of metals is considered in a generic sense in the industry to describe various operations performed on metals including drawing, stamping, rolling, forging, heading, extruding, cupping, forming, bending, seaming, curling, sizing, swaging, embossing, coining, spinning, punching, piercing, parting, shearing, splitting, lancing, trimming, shaving, broaching, drinking, burring, cutting, machining, grinding, etc. The specific method of working will vary with the individual machine shop, press shop, foundry, fabricator, etc., to suit its particular apparatus and requirements. It is understood that this term is used in a like manner in the present specification and claims to include one or more of the above operations.

In the working of metals it is important that certain steps be taken to insure efficient operation. Because the working operation involves the contact of two different metals (the die and the metal being worked), it is of importance to utilize a lubricant to avoid seizure, fusion, attrition of the metals, etc. Also, it is important to utilize a cooling medium to dissipate the heat of friction caused by the contact of the metals. Various liquids, pastes, flakes or compositions heretofore have been used to serve both as a lubricant and coolant, these being known in the industry as drawing compounds. The present invention provides a novel drawing compound which serves as an effective lubricant and coolant and, in addition, also serves to retard corrosion of the formed metal. This is important because the formed article generally is stored prior to shipment and may undergo corrosion during such storage or it may undergo corrosion during the shipment. Another important advantage to the use of the novel drawing compound of the present invention is that it also protects the die against corrosion which otherwise may occur, particularly while standing during shutdown. In addition, the novel drawing compound offers other important advantages, as will be hereinafter set forth.

The novel drawing compound of the present invention is used in the working of any suitable metal or alloy, including both ferrous and non-ferrous. Illustrative but not limiting examples include low-carbon steel, high-carbon steel, sheet steel, cast iron, aluminum, tungsten, magnesium, tin, copper, titanium, vanadium, nickel, platinum, silver, etc., and alloys including, for example, brass, bronze, etc. These may be formed into various parts including, for example, wire, tubing, automotive body parts such as doors, tops, cowl, frames, spring suspension parts, bumper brackets, bearings, wheels, brake bands, clutch plates, electrical contacts, etc., parts for metal furniture, refrigerator parts, machine parts, typewriter parts, adding machine parts, railroad and aircraft parts, handles for doors and drawers, etc., internal equipment for reaction chambers, fractionating columns, heat exchangers, pumps, etc. It is understood that these are but a few of the many diversified metal parts that are formed by working in the manner hereinafter set forth,

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and that the novel drawing compound of the present invention is used in any metal working operation.

While the novel drawing compound of the present invention can be used in some instances in the hot working of metals, it is particularly useful in cold working processes. In the latter operation, it is important that the drawing compound serves effectively as a lubricant and also as a coolant to dissipate the heat of friction created upon contact of the metals. Also, it is of extreme importance that the metals be protected against corrosion. In addition, as hereinbefore set forth, the novel drawing compound offers other important advantages to be described hereinafter.

In one embodiment the present invention relates to the method of working a metal which comprises effecting said working in the presence of a drawing compound containing (1) salt of amine having at least 8 carbon atoms and carboxylic acid having at least 6 carbon atoms and (2) salt of amine having at least 8 carbon atoms and alkyl acid phosphate in which at least 1 alkyl group contains at least 3 carbon atoms.

From the hereinbefore embodiment, it will be seen that the novel drawing compound contains a mixture of two different amine salts. This mixture of amine salts is hydrocarbon soluble and preferably is formed as a solution in a suitable hydrocarbon oil in the manner to be hereinafter set forth. In another embodiment the mixture of salts may be incorporated as an additive in conventional drawing compounds and serves to impart therein the important advantages herein set forth.

One component of the drawing compound of the present invention is an amine salt of a carboxylic acid. Any suitable amine may be employed and may comprise a primary monoamine, but preferably comprises a diamine and, still more particularly, an N-alkyl-diaminoalkane. Preferably, the amine contains at least 8 carbon atoms and, in general, will contain from about 12 to about 40 carbon atoms per molecule. Illustrative primary monoamines include octylamine, nonylamine, decylamine, undecylamine, dodecylamine, tridecylamine, tetradecylamine, pentadecylamine, hexadecylamine, heptadecylamine, octadecylamine, nonadecylamine, eicosylamine, etc. The amine may be prepared from fatty acid derivatives and thus may comprise tallow amine, hydrogenated tallow amine, lauryl amine, coconut amine, soya amine, etc.

Of the diamines, the N-alkyl-diaminoalkanes are preferred. A particularly preferred amine of this class comprises an N-alkyl-1,3-diaminopropane in which the alkyl group contains from about 8 to about 25 carbon atoms. A number of N-alkyl-1,3-diaminopropanes of this class are available commercially, such as "Duomeen T" and "Diam 26" in which the alkyl group is derived from tallow and contains from about 12 to about 20 carbon atoms per group, and mostly 16 to 18 carbon atoms. Other N-alkyl-1,3-diaminopropanes may be prepared to contain any number of carbon atoms desired in the alkyl group and thus the alkyl group is selected from hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, nonadecyl, eicosyl, etc.

While the N-alkyl-1,3-diaminopropanes are preferred, it is understood that other suitable N-alkyl-diaminoalkanes may be employed. Illustrative examples include N-alkyl-1,2-diaminoethane, N-alkyl-1,2-diaminopropane, N-alkyl-1,2-diaminobutane, N-alkyl-1,3-diaminobutane, N-alkyl-1,4-diaminobutane, N-alkyl-1,2-diaminopentane, N-alkyl-1,3-diaminopentane, N-alkyl-1,4-diaminopentane, N-alkyl-1,5-diaminopentane, N-alkyl-1,2-diaminohexane, N-alkyl-1,3-diaminohexane, N-alkyl-1,4-diaminohexane, N-alkyl-1,5-diaminohexane, N-alkyl-1,6-diaminohexane,

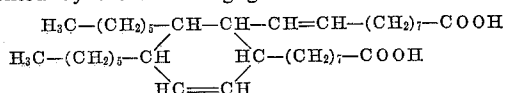
etc. It is understood that a mixture of amines may be employed and also that the different amines are not necessarily equivalent but all of them may be used in the preparation of active compositions.

As hereinbefore set forth, the amine salt of a carboxylic acid is used as one component of the mixture. Any suitable carboxylic acid is used, preferably a polybasic carboxylic acid. The carboxylic acid preferably contains at least 6 and still more preferably at least 10 carbon atoms per molecule and, more particularly, from about 20 to about 50 carbon atoms per molecule. Illustrative polybasic carboxylic acids include adipic, pimelic, suberic, azelaic, sebacic, phthalic, etc., aconitic, citric, etc., hemimellitic, trimelic, prehnitic, mellophanic, pyromellitic, mellitic, etc., and higher molecular polybasic carboxylic acids. It is understood that a mixture of acids may be employed.

A particularly preferred acid comprises a mixed by-product acid being marketed commercially under the trade name of "VR-1 Acid." This acid is a mixture of polybasic acids, predominantly dibasic, and is a residue produced by distilling, at about 270° C. under about 4 mm. of mercury pressure, the by-product acids obtained in the preparation of sebacic acid by fusing castor oil with alkali. Production of this residue is described in more detail in U.S. Patent 2,267,269 to Cheetham et al. In the manufacture of sebacic acid from castor oil, the oil is heated with a caustic alkali. This splits the oil, forming octanol-2, methylhexyl ketone, the alkali salt of sebacic acid, and the alkali salts of various other long-chained acids. The alcohol and ketone are readily removed from the reaction mixture by distillation. The alkali salts which remain then are dissolved in water and, upon slight acidification of the resulting solution, an oily layer separates. At a pH of about 6, the aqueous phase contains the alkali salt of sebacic acid, while the oily layer contains various other acids from the reaction. The term "by-product acids" is generally applied to the mixture of acids forming the oily layer.

These by-product acids then are separated into two parts. After these acids have been washed with a dilute mineral acid, such as sulfuric or hydrochloric, they are washed with water and dried. They then are distilled under reduced pressure. Fatty acids which are primarily monobasic carboxylic acids are taken off at 100° C. to 270° C. at pressures as low as 4 mm. This treatment leaves a residue which is a mixture of fatty acids, apparently primarily polybasic in character. The residue is commercially available from Rohm & Haas Company under the trade name of "VR-1 Acid" and has an average molecular weight of 500-600, an acid number of 134-160, a saponification number of 174-179, and an iodine number of 53-60. Another mixed acid of substantially the same composition is available commercially under the trade name of "D5OME Acid."

Another preferred acid comprises a mixed acid being marketed commercially under the trade name of "Empol 1022." This dimer acid is a dilinoleic acid and is represented by the following general formula:



This acid is a viscous liquid, having an apparent molecular weight of approximately 600. It has an acid value of 180-192, an iodine value of 80-95, a saponification value of 185-195, a neutralization equivalent of 290-310, a refractive index at 25° C. of 1.4919, a specific gravity at 15.5° C./15.5° C. of 0.95, a flash point of 530° F., a fire point of 600° F., and a viscosity at 100° C. of 100 centistokes.

While the polycarboxylic acids generally are preferred, in another embodiment of the invention a monocarboxylic acid may be used or a mixture of a monocarboxylic acid and the polycarboxylic acid hereinbefore set forth.

Here again, it is preferred that the carboxylic acid contains at least 6 atoms per molecule and may range up to 50 carbon atoms per molecule. Illustrative monocarboxylic acids include caproic, heptylic, caprylic, pelargonic, capric, lauric, myristic, palmitic, stearic, arachidic, behenic, lignoceric, cerotic, etc., decalenic, dodecalenic, palmitoleic, oleic, ricinoleic, petroselinic, vaccenic, linoleic, linolenic, eleostearic, licanic, parinaric, gadoleic, arachidonic, cetoleic, erucic, selacholeic, etc.

Here again, mixtures of such acids are available commercially at lower cost and advantageously are used in preparing the drawing compound of the present invention. A particularly preferred mixture is tall oil acid which comprises a mixture of saturated and unsaturated fatty acids and rosin acids and is obtained by acidifying the black liquor skimmings obtained in the pulping of wood. The manufacture of tall oil acid is described in the Encyclopaedia of Chemical Technology, volume 13, pages 572-577. A number of typical American tall oil acids are shown on page 575 of this encyclopedia, along with typical properties thereof.

While the crude tall oil acid may be used in accordance with the present invention, it is preferable to use a purified tall oil acid. A particularly satisfactory tall oil acid is available commercially under the trade name of "Indusoil L-5." This acid has the following typical properties:

Specifications	Minimum	Maximum
Color, Gardner		7
Acid No.	188	196
Saponification No.	190	198
Fatty acids, percent	90	
Rosin acids, percent		5
Unsaponifiables, percent		4
Specific gravity, 60F/60F	0.905	0.910
Pour Point, ° F.	45	55
Flash Point, Open Cup, ° F.	360	400
Fire Point, Open Cup, ° F.	410	440

When a mixture of the monocarboxylic acid and the polycarboxylic acid is used, both being selected from those hereinbefore set forth, it generally is preferred to use these in a ratio of one equivalent of each with two equivalents of the amine. However, in some cases, an excess of one acid may be employed and thus may range up to ten or more equivalents of the monocarboxylic acid with one equivalent of the polycarboxylic acid, or up to ten or more equivalents of the polycarboxylic acid with one equivalent of the monocarboxylic acid.

The amine salt of carboxylic acid is prepared in any suitable manner. In general, the salt is readily prepared by admixing the amine and carboxylic acid at ambient temperature, preferably with vigorous stirring. Elevated temperature may be employed, but generally will not exceed about 250° F. When desired, the salt may be prepared in the presence of a solvent for ease in handling, either of the amine and/or acid of the final mixture. Any suitable solvent may be employed and may comprise an aromatic hydrocarbon such as benzene, toluene, xylene, ethylbenzene, etc., or a mixture such as naphtha, which may be straight run, catalytically cracked, thermally reformed, catalytically reformed, preferably in the presence of hydrogen or mixtures thereof, kerosene, lubricating oil, etc. Depending upon the particular drawing composition in which the mixture is employed, the lower boiling solvent may be removed by fractionation or it may be allowed to remain in the mixture. When desired, the mixture will be marketed as a solution in a suitable solvent and the same solvent used in the final mixture also may be used during the preparation of the amine-carboxylic acid salt.

In general, the neutral salt of the amine and carboxylic acid is preferred. The neutral salt is prepared by utilizing stoichiometric amounts of the carboxylic acid and amine. In other words, the concentrations of the car-

boxylic acid and amine are selected so that there is an equivalent number of carboxylic acid groups to amine groups. In another embodiment the salt may be a basic salt, which is prepared by utilizing a deficiency of carboxylic acid groups in relation to the amino group as, for example, by utilizing one equivalent of carboxylic acid per two equivalents of amine. In still another embodiment, an acid salt may be employed, which may be prepared by using an excess of acid with relation to the amine as, for example, two equivalents of acid per one equivalent of amine. Accordingly, the ratio of amine to carboxylic acid may range from 0.2:1 to 5:1. It is understood that these different salts are not necessarily equivalent.

As hereinbefore set forth, another component of the drawing compound of the present invention is an amine salt of an alkyl acid phosphate. The amine preferably is selected from the amines hereinbefore specifically set forth in the description of the amine salt of carboxylic acid. In the interest of simplicity, these amines are not repeated here, but it is understood that the amine is selected from those hereinbefore specifically set forth.

In one embodiment, the amine used in the preparation of the salt of the alkyl acid phosphate is the same amine as used in the preparation of the amine salt of the carboxylic acid. In another embodiment, a different amine is used in the preparation of each of these salts, but the amines preferably are selected from those hereinbefore specifically set forth.

Any suitable alkyl acid phosphate is used in preparing the salt. The term "alkyl acid phosphate" includes both the alkyl acid orthophosphates and the alkyl acid pyrophosphates. In the alkyl acid orthophosphates, the mono-alkyl ester, dialkyl ester or a mixture thereof may be employed. In the alkyl acid pyrophosphate, the mono-alkyl ester, dialkyl ester, trialkyl ester or a mixture thereof may be employed, the dialkyl ester being preferred and the ester groups being attached to the same or different phosphorus atom. Generally, however, this compound will be symmetrical and, thus, the alkyl ester groups will be attached to different phosphorus atoms.

At least one of the alkyl groups constituting the ester portion of the alkyl acid phosphate contains at least 3 and preferably at least 5 carbon atoms and may contain up to 20 and preferably up to 12 carbon atoms. Illustrative examples of preferred alkyl acid orthophosphates include monoamyl acid orthophosphate, diamyl acid orthophosphate, monohexyl acid orthophosphate, dihexyl acid orthophosphate, monoheptyl acid orthophosphate, diheptyl acid orthophosphate, monooctyl acid orthophosphate, dioctyl acid orthophosphate, monononyl acid orthophosphate, dinonyl acid orthophosphate, monodecyl acid orthophosphate, didecyl acid orthophosphate, monoundecyl acid orthophosphate, diundecyl acid orthophosphate, monododecyl acid orthophosphate, didodecyl acid orthophosphate, etc. As hereinbefore set forth, these are the preferred alkyl acid phosphates. However, it is understood that the alkyl group or groups may contain from about 3 to about 20 carbon atoms.

Preferred alkyl acid pyrophosphates include monoamyl acid pyrophosphate, diamyl acid pyrophosphate, monohexyl acid pyrophosphate, dihexyl acid pyrophosphate, monoheptyl acid pyrophosphate, diheptyl acid pyrophosphate, monooctyl acid pyrophosphate, dioctyl acid pyrophosphate, monononyl acid pyrophosphate, dinonyl acid pyrophosphate, monodecyl acid pyrophosphate, didecyl acid pyrophosphate, monoundecyl acid pyrophosphate, diundecyl acid pyrophosphate, monododecyl acid pyrophosphate, didodecyl acid pyrophosphate, etc. Here again, it is understood that the alkyl pyrophosphates may contain from about 3 to about 20 carbon atoms in the alkyl group or groups.

Alkyl acid phosphates are manufactured commercially as a mixture of the mono- and di-alkyl acid phosphates and such mixtures generally are available at a

lower cost. As another advantage of the present invention, such lower cost mixtures may be used in preparing the drawing compound of the present invention. Particularly preferred mixed alkyl acid phosphates comprise a mixture of mono- and di-isomyl acid orthophosphate, a mixture of mono- and di-isooctyl acid orthophosphate and the corresponding mixed alkyl acid pyrophosphates.

The amine-alkyl acid phosphate salt is prepared in any suitable manner. In general, this salt is prepared in substantially the same manner as the amine-carboxylic acid salt as hereinbefore described. The amine and alkyl acid phosphate are commingled, either at ambient temperature or slightly elevated temperature, with vigorous stirring. When an elevated temperature is employed, it generally will not exceed about 250° F. Here again, a solvent may be used for ease in handling, the solvent preferably being selected from those hereinbefore specifically set forth.

In general, the neutral salt of the alkyl acid phosphate and amine is preferred. The neutral salt is prepared by utilizing stoichiometric amounts of the alkyl acid phosphate and the amine. In other words, the concentration of alkyl acid phosphate and amine will be selected so that there will be an equivalent number of acid groups to amino groups. Thus, the specific concentrations will depend upon whether the orthophosphate or pyrophosphate salt is prepared and whether a monoamine or polyamine is used. In another embodiment, the salt is a basic salt, which is prepared by utilizing a deficiency of acid groups in relation to the amino group as, for example, by utilizing one equivalent of acid per two equivalents of amine. In still another embodiment, an acid salt is employed, which is prepared by using an excess of acid with relation to the amine as, for example, two equivalents of acid per one equivalent of amine. Accordingly, the ratio of amine to alkyl acid phosphate may range from 0.2:1 to 5:1. It is understood that these different salts are not necessarily equivalent.

In the preparations described above, the amine salt of the carboxylic acid and the amine salt of the alkyl acid phosphate are separately formed and then are commingled in the desired proportions. In another embodiment, the amine salts may be prepared in a single step. In this embodiment, the carboxylic acid and the alkyl acid phosphate are commingled, preferably with vigorous stirring, and then the amine or amines are added thereto with vigorous stirring. The reaction is effected at ambient temperature, or an elevated temperature which generally will not exceed about 250° F. may be employed. In cases where the amine is a solid at ambient temperature, the amine may be heated up to about 250° F. in order to melt the same and the melted amine then is added to the acids as aforesaid.

The salt of carboxylic acid and the amine salt of alkyl acid phosphate are viscous liquids when prepared in the absence of solvents. Accordingly, for convenience in handling and also for economic reasons, it is preferred that the active ingredients be used in the form of a solution in a suitable solvent and particularly mineral oil. When one or both of the salts are prepared in the presence of a solvent, preferably the solvent is retained in the composition and, when desired, additional solvent is added to form a composition of the desired consistency. In the cold working process, a low boiling, intermediate boiling or high boiling solvent may be used, depending upon the particular working operation. In the hot working operation, a high boiling solvent preferably is employed in order to avoid vaporization of the solvent and possible fire hazard. As hereinbefore set forth, the consistency of the drawing composition will vary with each particular machine shop, press shop, foundry, fabricator, etc., to suit its particular requirements.

The amine salt of carboxylic acid and the amine salt of alkyl acid phosphate generally are used in substantially equal concentrations, but may be employed in propor-

tions ranging from 0.1 to 10 and particularly from 0.5 to 2 parts by weight of one salt per 1 part by weight of the other salt. The concentration of total active ingredient (total of amine salt of carboxylic acid and of amine salt of alkyl acid phosphate) in the final drawing composition may vary considerably. In general, this concentration will be in the range of from about 0.1% to about 50% and preferably from about 0.5% to about 20% by weight of the final composition.

In one embodiment the amine salts are prepared as a concentrate of active ingredients and will be dissolved in a solvent in the desired proportions. In another embodiment the amine salts are prepared as a concentrated solution in a solvent, and then are diluted further with additional solvent to the desired concentration. As hereinbefore set forth, the consistency of the final drawing composition will vary with the particular user to suit his peculiar requirements.

In another embodiment of the invention, the concentrated active ingredients or a concentrated solution thereof may be added to a conventional drawing compound. A large number of conventional drawing compounds were investigated by the present applicants and found to impart little or no protection from corrosion. When the active ingredients of the present invention are incorporated in such drawing compounds, the final drawing compound will afford protection against corrosion and also will offer the additional advantages hereinafter set forth. In this embodiment, the drawing compound should contain the active ingredients of the present invention in a total concentration of from about 0.5 to about 20% by weight of the final composition.

The drawing compound of the present invention is used in any suitable manner and generally as conventionally used in the shops. This may comprise passing the metal to be worked through a layer of the drawing compound, thereby forming a film of drawing compound on the metal, and then passing the metal into the working operation. In another embodiment a flowing stream of the drawing composition may be passed over the metal and/or die before or during the working operation. No novelty is claimed herein for any particular method of using the drawing compound and, as hereinbefore set forth, any suitable method may be employed.

Regardless of the method of applying the drawing compound, the novel composition of the present invention offers important advantages in the working operation. As hereinbefore set forth, this composition is an effective lubricant and also an effective coolant. In addition, the novel drawing compound of the present invention serves to impart corrosion protection to both the metal being worked and the die used in the operation. As hereinbefore set forth, imparting protection from corrosion to the metal is important in the further use or working thereof. Additionally, the drawing compound protects the die from corrosion, either during use or during periods of shutdown. It is a considerable contribution to the field of drawing compounds that all three of these important requirements are obtained through the use of a single composition. This avoids the additional time and expense heretofore required in utilizing two different compositions and two different treatments to obtain these results.

As another important advantage to the novel drawing compound of the present invention, it has been found that the heretofore necessity of periodically dressing a die for shallow drawing of close tolerance appears to be eliminated. In other words, heretofore in such operations, it has been necessary to periodically buff the die with an emery cloth to rub out rough spots and scratches. However, when using the novel drawing compound of the present invention, the dressing operation no longer is necessary and, in fact, it appears that the die actually seems to gain in polish.

As another important advantage to the use of the novel drawing compound of the present invention, it appears to

considerably reduce "blow back" normally encountered in subsequent casting operations. "Blow back" apparently is caused by deposits on objects which are being subjected to casting. However, it has been found that metal parts formed in the presence of the drawing compound of the present invention apparently contain very little or no deposits because "blow back" is no problem during subsequent casting operations. Therefore, when the metal part formed in the presence of the drawing compound of the present invention is joined to another part in a casting operation, the reduction or elimination of "blow back" during the casting operation is another important advantage.

As still another advantage to the novel drawing compound of the present invention, it has been found that there are no objectionable effects to the weldability of the formed metal. Therefore, welding of the metal is readily accomplished when desired. Still another advantage is that the metal part may be painted without objectionable effects. As still another advantage, it appears that the use of the drawing compound of the present invention results in a cleaner finished part and, therefore, may eliminate subsequent cleaning which otherwise may be required when using other drawing compounds.

From the above description, it will be seen that the novel drawing compound of the present invention offers many advantages over the drawing compounds presently being used. Of utmost importance is the fact that lubrication, cooling and corrosion prevention are obtained in a single operation, thereby avoiding the heretofore necessity of requiring two separate operations to accomplish this. In addition, other important advantages are obtained including protecting the die from corrosion, avoiding "blow back," eliminating periodic dressing of the dies, eliminating cleaning of the formed articles, etc.

The following examples are introduced to illustrate further the novelty and utility of the present invention but not with the intention of unduly limiting the same.

Example I

The active ingredients of the drawing compound of this example comprised 50% by weight of the neutral salt of "Duomeen T" and "D5OME Acid" and 50% by weight of the neutral salt of "Duomeen T" and mixed mono- and diisooctyl acid orthophosphates. As hereinbefore set forth, "Duomeen T" is N-tallow-1,3-diaminopropane and contains predominantly 16 to 18 carbon atoms in the tallow group. Also, as hereinbefore set forth, "D5OME Acid" is of substantially the same composition as "VR-1 Acid." The mixed mono- and diisooctyl acid orthophosphates are available commercially.

For ease in handling, the preparation was made in admixture with a commercial paraffinic oil having an API gravity at 60° F. of about 28° and a viscosity at 100° F. of about 108 SUS. The neutral salts were prepared in a single step operation by dissolving 360 pounds (1 equivalent weight) of "D5OME Acid" in 500 pounds of the paraffinic oil and vigorously mixing at ambient temperature. 250 pounds (1 equivalent weight) of the mixed mono- and diisooctyl orthophosphates then were added with continued mixing. Finally, 346 pounds (2 equivalent weights) of "Duomeen T" were added and the vigorous mixing continued until a homogeneous solution was formed. The concentrate prepared in the above manner then was diluted with additional paraffinic oil to form a solution containing the active ingredients in a total concentration of 40% by weight.

The concentrated solution described above was diluted further in a proportion of 1 volume of the concentrated solution and 5 volumes of commercial paint thinner. The final solution was used as a drawing compound for the drawing of cold roller steel to form a hub seal. This operation is drawn to a close tolerance and therefore requires a very effective drawing compound. A steel disc of 7" diameter was stamped to form a hub seal of 5" diameter having a peripheral flange of 3/4". The metal

disc was dipped into a solution of the drawing compound described above prior to the stamping operation.

As hereinbefore set forth, numerous commercial drawing compounds were investigated by the present applicants and it was found that none of these conventional drawing compounds afforded corrosion protection to the finished article. When stored outside, the finished article formed in the presence of the conventional drawing compounds began rusting within 5 days.

In contrast to the above, the hub seal formed in the presence of the drawing compound of the present invention described above did not show any signs of corrosion after more than two months of outdoor storage.

In addition to permitting the close tolerance drawing, the drawing compound of the present invention protected the die from corrosion during shutdown. Heretofore, it had been necessary to periodically dress the surface of the die. This dressing operation consisted of using an emery cloth and rubbing out rough spots and scratches. When using the drawing compound of the present invention, the dressing operation was unnecessary and, in fact, the die seemed to gain in polish.

Example II

The drawing compound of this example was prepared in the manner described in Example I except using a 1:1 volume dilution with commercial paint thinner. This drawing compound was used in the cold forming of a part to be integrated with a cast part. As hereinbefore set forth, during the casting a serious problem is the "blow back" apparently caused by deposits on the object being cast. Surprisingly, it was found that "blow back" was practically eliminated when using the part formed by cold rolling in the presence of the drawing compound of the present invention.

Example III

The concentrated solution, prepared as described in Example I, also was added to a commercial drawing compound. The concentrated solution was added in a proportion of 1 volume of concentrated solution per 5 volumes of the commercial drawing compound.

When used for the forming operation described in Example I, it was found that the hub seal formed in the presence of this drawing compound underwent no corrosion for a period of two months in outdoor storage. In contrast, the hub seal formed in the presence of the commercial drawing compound (not containing the concentrated solution) underwent corrosion within 5 days of outdoor storage.

Example IV

The drawing compound of this example comprises 5% by weight of the neutral salt of tallow amine and tall oil acid, 5% by weight of the neutral salt of tallow amine and mixed mono- and di-isoamyl acid orthophosphates, and 90% by weight of a commercial paraffinic oil. The salt of tallow amine and tall oil is prepared by mixing these materials at room temperature with vigorous stirring. The salt of tallow amine and mixed mono- and di-isoamyl acid orthophosphates is separately prepared by mixing these materials at room temperature with vigorous stirring. The salts then are commingled with the paraffinic oil in the proportions described above.

The solution described above is used as a drawing compound to form small motor cases from cold rolled coil

stock. The drawing compound is roller-coat applied to the metal prior to forming. Following the forming operation, it is found that the parts do not need to be cleaned and that they will remain free from corrosion when stored outside prior to further processing.

We claim as our invention:

1. The method of working a metal which comprises effecting said working in contact with a drawing composition consisting essentially of from about 0.1% to about 50% by weight of a mixture of (1) a salt of an amine having at least 8 carbon atoms and a carboxylic acid having at least 6 carbon atoms and (2) a salt of an amine having at least 8 carbon atoms and an alkyl acid phosphate in which at least 1 alkyl group contains at least 3 carbon atoms, said salts being free of metal and being in the proportion of from about 0.1 to 10 parts by weight of one of the salts per 1 part by weight of the other salt.

2. The method of claim 1 wherein said salts are dissolved in a mineral oil in a concentration of from about 0.1 to about 50% by weight of the final composition.

3. The method of claim 1 wherein said salts are dissolved in a mineral oil in a concentration of from about 0.5 to about 20% by weight of the final composition.

4. The method of claim 1 wherein said salts are (1) a salt of a diamine having from 8 to about 40 carbon atoms per molecule and a carboxylic acid having from 6 to about 50 carbon atoms per molecule and (2) a salt of a diamine having from 8 to about 40 carbon atoms per molecule and an alkyl acid phosphate in which at least 1 alkyl group contains from about 3 to about 20 carbon atoms.

5. The method of claim 1 wherein said salts are (1) a salt of N-alkyl-1,3-diaminoalkane in which said alkyl contains at least 8 carbon atoms and a dicarboxylic acid having at least 6 carbon atoms and (2) a salt of N-alkyl-1,3-diaminoalkane in which said alkyl contains at least 8 carbon atoms and an alkyl acid phosphate in which at least 1 alkyl group contains at least 3 carbon atoms.

6. The method of claim 1 wherein said salts are (1) a salt of N-tallow-1,3-diaminopropane and a dicarboxylic acid having from about 10 to about 50 carbon atoms and (2) a salt of N-tallow-1,3-diaminopropane and mixed mono- and di-alkyl acid orthophosphates.

7. The method of claim 6 wherein said mixed phosphates are mixed mono- and di-amyl acid orthophosphates.

8. The method of claim 6 wherein said mixed phosphates are mixed mono- and di-octyl acid orthophosphates.

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MARCUS U. LYONS, *Primary Examiner*.

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