


5-1-1939

A Study of Solders

John D. Prater

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Prater, John D., "A Study of Solders" (1939). *Bachelors Theses and Reports, 1928 - 1970*. Paper 100.

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A STUDY OF SOLDERS

By

John D. Prater

A Thesis
Submitted to the Department of Metallurgy
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Metallurgical Engineering

MONTANA SCHOOL OF MINES
BUTTE, MONTANA
May, 1939

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W/496-145933

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A STUDY OF SOLDERS

INTRODUCTION

The introduction of the use of solders was probably made in late Roman times. According to Pliny¹, two compositions were in common use at this time: "tertiarium," consisting of one part of tin and two of lead; and "argentarium," consisting of equal parts of the two metals. It is interesting to note that these solders are of the same composition as those widely used today; "tertiarium" corresponding to plumbers' solder and "argentarium" of the same composition as tinner's solder.

From Roman times until the present century, only one important introduction was made in the soldering process. This came with the introduction of the wiped joint in 1432, when it was used in installing the Bayswater lead conduit. The present century marks the beginning of a new era in the soldering field with the introduction of machine soldering.

Indicative of the importance of solders in our present civilization is the vast amount of lead and tin which is consumed annually in their production. In 1936, the latest year for which figures are available, 22,000 short tons of lead and 20,500 short tons of tin were consumed.²

The wetting of liquids by liquids has been a subject of considerable study, and the phenomenon is fairly well understood both quantitatively and qualitatively. Chemical-

1. MacNaughtan, D. J., and Ernest S. Hedges: Solder. International Tin Research and Development Council, Bulletin 2, P. 4 (September, 1935)

2. U. S. Bureau of Mines: "Minerals Yearbook" U. S. Gov't. Printing Office, Washington (1938); PP. 119 and 617.

ly, it is explained by the attraction of the molecules of one liquid for those of the other. The wetting of solids by liquids is also explained by the chemical affinities of the atoms or molecules at the surface. Little qualitative work has been done in this field, due to the difficulty of measuring the surface energy of solids and that of liquid-solid interfaces.³

The purpose of this paper is to furnish a qualitative method of comparing the degree of wetting of various solders upon different plate metals when several fluxes are used.

EXPLANATION OF TERMS

Solder is a fusible metal or alloy used to unite other metals or alloys. Important properties of a solder are a low melting point and a ready adhesion for other metals. As used in this paper, the term will refer only to alloys of lead and tin. It cannot be considered a specific substance but comprises a whole series of alloys, the properties of which vary with the composition.

Plate metal is the term referring to the metals which are to be joined together.

Flux is a substance which is used to insure perfect adhesion between the solder and metal. The function of a flux is twofold: (1) to protect the surface from oxidation at the temperature required for soldering, and (2) to remove existing oxide or dirt from the surface.

3 Daniels, E. M., and D. J. MacNaughtan: Wetting of Metals by Metals with Particular Reference to Tinning and Soldering, Technical Publications of the International Tin Research and Development Council. Series B. No. 6, P. 3 (Feb., 1937)

Wetting is that phenomenon which causes a drop of liquid to spread indefinitely over a solid when the liquid is placed in contact with the surface. The solid is said to be unwetted by the liquid if the liquid comes to rest in the form of a globule. Crow⁴ suggests that if wetting occurs between two metals, alloying invariably follows; and conversely, alloying must be preceded by wetting.

The degree of wetting is measured by the contact angle. Reinders⁵ calculated that the cosine of the angle made by the adhering bubble and solid surface is related to the surface tensions involved as follows (see Fig. 1):

$$\cos \theta = \frac{T_{SG} - T_{SL}}{T_{LG}}$$

in which T_{SG} is the surface tension between the

solid and gas.

T_{SL} is the surface tension between the

solid and liquid.

T_{LG} is the surface tension between the

liquid and gas.

θ is the supplement of the contact angle.

The contact angle thus provides a convenient method for measuring the efficacy of solders.

⁴ Crow, T. B.: Fluxes for Soft Soldering, Reprint from Trans. Faraday Society, Vol. XX, P. 200 (Part I, August, 1924)

⁵ Ralstron, Oliver C.: Why Do Minerals Float, Min. Sci. Press, 111, 623-27 (1915)

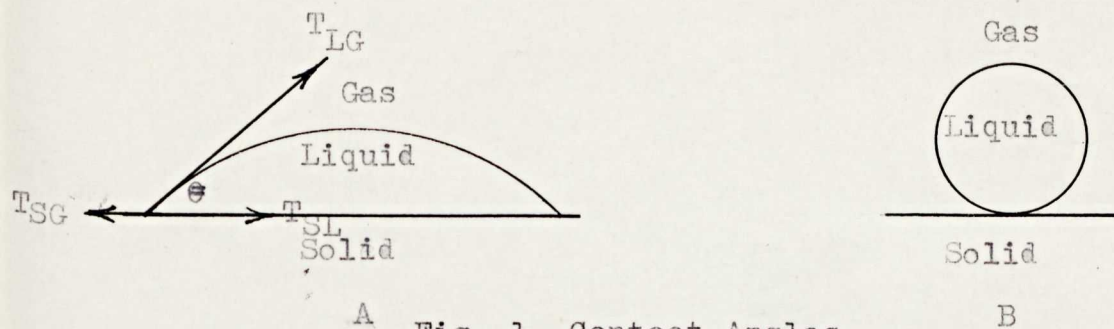


Fig. 1. Contact Angles

A, liquid wets metal; B, liquid does not wet metal.

PROPERTIES OF SOLDERS

Much regarding the properties of *solders* can be learned from a study of the equilibrium diagram shown in Figure 2.⁶ With the exception of the alloy containing 61.9 per cent tin, the solidification occurs over a range of temperature. The alloy containing 61.9 per cent tin has the lowest melting point of the series and solidifies sharply. The other solders can be worked since they are in a pasty condition over a range of temperature. The temperature of complete liquefaction and complete solidification of the solders investigated are shown in Table I.

Table I. Properties of Solders.

Solder Composition		Temperature of:	
Pb	Sn	Complete Solid.	Comp. Liq.
33%	67%	183.3° C.	190° C.
50%	50%	183.3° C.	220° C.
67%	33%	183.3° C.	260° C.

⁶ Hansen, Von M.: "Der Aufbau Zweistofflegierungen,"

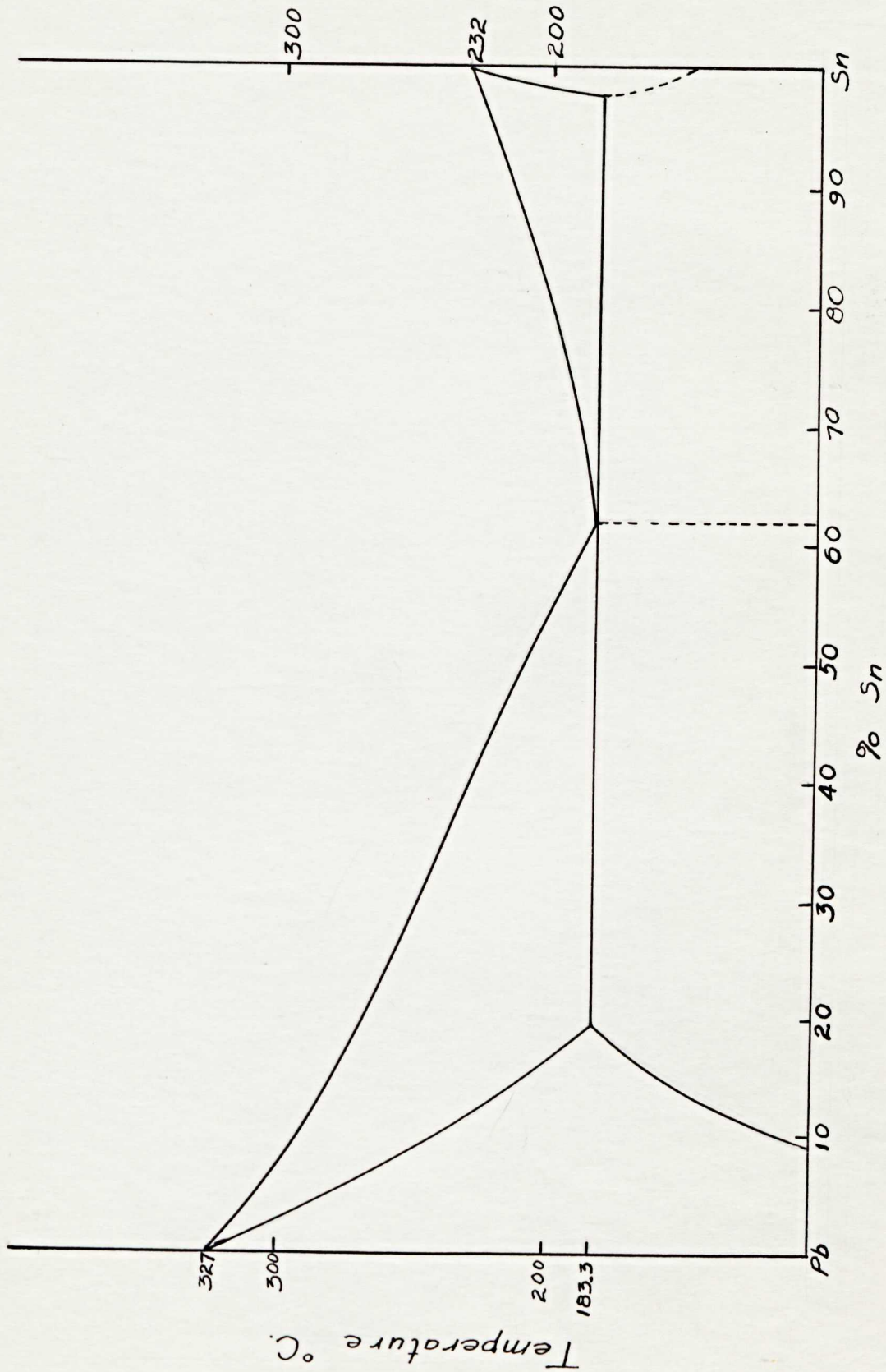


Figure 2. Pb-Sn Diagram

Practically all commercial solders fall within the range 16 to 61.9 per cent tin. Therefore, the solidified commercial solders consist of crystals of lead surrounded by eutectic. As the tin content is increased within this range, the relative proportion of eutectic to free lead is also increased. As the composition nears that of the eutectic, the freezing range becomes correspondingly smaller. As shown in the diagram, tin is soluble in lead up to about nine per cent at 100 C., but lead is only very slightly soluble in tin.

EXPERIMENTATION

The alloys of tin and lead were prepared over a Bunsen flame in an unglazed crucible lined with flaked graphite to prevent excess oxidation of the metals during melting. The lead was first melted and the tin added. The solders thus prepared were of the approximate composition: (1) two parts of tin to one of lead, (2) equal parts of the two metals, and (3) two parts of lead to one of tin. It is seen that the first solder is near the eutectic composition while the second and third are on the lead rich side of the diagram.

Table II. Solders Prepared

Weight of Pb.	Weight of Sn.	Weight of resulting solder
15.0 g.	7.5 g.	21.5 g.
16.7 g.	16.7 g.	25.8 g.
30.0 g.	15.0 g.	38.1 g.

The plate metals copper, aluminum, nickel, zinc, iron, and nevastain were each used to determine the degree of wetting which was obtained when given different preliminary treatment and treated with various fluxes. The fluxes used were ammonium chloride, zinc chloride, a mixture of equal parts of zinc and ammonium chlorides, and rosin. The solder used in the preliminary experiments was that containing two parts of lead and one part of tin.

Series 1. The plate metal was cleaned with number 0 emery paper and heated on a hot plate to about 250° C. The solder was melted on with a Bunsen burner, touched with the flux, and the effect noted.

Series 2. The plate metal was uncleaned, but with this exception was treated the same as Series 1.

Series 3. The plate metal was cleaned with number 0 emery paper and placed on the hot plate with the flux and solder in contact. The three were heated together and the effect noted as before.

Series 4. The plate metal was treated similar to Series 3, except that it was not cleaned previous to being placed on the hot plate.

The detailed results of these experiments follow.

In no case did wetting occur when the solder was melted on the hot plate metal until the solder was touched with the flux. The globule of solder remained as an oxidized blob which could easily be brushed off leaving no trace. In all cases, however, the solder was cleaned to a bright shining

globule immediately when touched with any of the fluxes.

With either chloride alone or with the mixture of the chlorides, the solder wetted the copper, nickel, and zinc and formed a good union with the plate metal. When nevastain was used as a plate metal, the zinc chloride alone or the mixture of zinc and ammonium chlorides caused a good union to be formed, but wetting was very slight when ammonium chloride was used alone.

In no case was rosin as effective a flux as any of the inorganic salts used. With copper, the wetting was so slight that the solder could be flicked off with the fingers. When nevastain was used, no union was effected; the globule would slide off when the plate was tipped. A union was effected with the other plate metals but in none, with the possible exception of zinc, did the spreading occur to so sufficient an extent as to even consider rosin as an effective flux under the conditions maintained. The inorganic fluxes caused a greater degree of wetting with zinc, so this would preclude rosin as a probable flux in this case. The aluminum was not wetted by the solder when any of the fluxes were used.

In general, the results obtained with the second series paralleled those of the first. The time required for wetting to take place was about one and one-half times as long as that necessary for the first group, and in no case did the solder wet the plate metal to as great an extent as it did when the latter had been previously cleaned. This was particularly noticeable when iron was used. Rosin did not form an effective union with any plate metal except zinc.

Best results were obtained with Series 3 when the flux

and solder were placed in contact with the cleaned plate metal before the latter was heated. Presumably, this is due to the fact that the flux protects the cleaned surface from oxidation while it is being heated. The solder remained as a free clean globule for a few seconds, then suddenly spread when the melting point of the solder was reached. With all plate metals except aluminum wetting occurred with each of the four fluxes. No flux caused wetting of the aluminum.

When the inorganic salts were used as fluxes and the surface of the plate metal was not cleaned, the results paralleled those of Series 3. The one notable exception was with neva-stain as the plate metal in which case ammonium chloride did not cause wetting to take place. The degree of wetting was decidedly reduced in all cases when rosin was used as a flux. Aluminum was not wetted by the solder when any of the fluxes were used.

In order to furnish a qualitative method of comparison, contact angles were measured. In addition to the fluxes used in the preceding experiments, a one per cent solution of sodium sulphide was also used. Three solders of the approximate composition two parts of lead to one of tin, one part of lead to one of tin, and one part of lead to two of tin, respectively, were used with each flux and with each plate metal. In the preceding experiments the best results were obtained when the flux and the solder were placed in contact with the plate metal before the latter was heated. Hence, in all tests where the angle of contact was to be measured, this procedure was followed. Three types of surface preparation for each plate metal were included in the

investigation; namely, oxidized, emiered, and emiered and etched.

The apparatus used for measuring the contact angle is shown in Figure 3. It consists essentially of a light with suitable lenses to focus and project the image on a screen

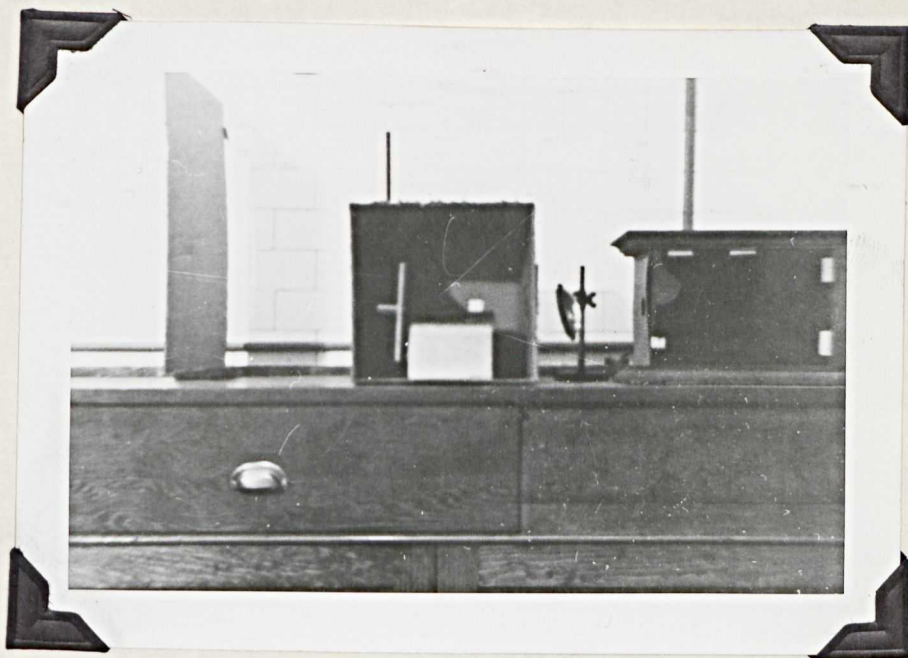


Fig. 3. Apparatus for measuring contact angle

at suitable magnification. The angle thus projected is measured by means of a protractor. In all cases where possible the contact angle formed at each side of the bubble was determined and the mean taken. The results are shown in Tables III to VII.

Table III a.
 Contact Angles with Copper
 Surface Oxidized to CuO

Solder Used	Flux used	Average Contact Angle
Pb = 33% Sn = 67%	Rosin	151° 15'
	Na ₂ S	151° 30'
	NH ₄ Cl / ZnCl ₂	170° 30'
	NH ₄ Cl	172° 30'
	ZnCl ₂	172° 30'
Sn = 50% Pb = 50%	NH ₄ Cl	158°
	ZnCl ₂	158°
	Rosin	165°
	Na ₂ S	168° 30'
	NH ₄ Cl / ZnCl ₂	180°
Pb = 67% Sn = 33%	NH ₄ Cl	154° 30'
	Na ₂ S	158° 15'
	ZnCl ₂	160°
	Rosin	167°
	NH ₄ Cl / ZnCl ₂	175°

Table III b.

Contact Angles with Copper
Surface Oxidized to Cu_2O

Solder used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Na_2S	45°
	NH_4Cl	146°
	Rosin	$162^\circ 30'$
	ZnCl_2	173°
	$\text{NH}_4\text{Cl} / \text{ZnCl}_2$	180°
Pb = 50% Sn = 50%	Na_2S	33°
	NH_4Cl	$157^\circ 45'$
	Rosin	$160^\circ 15'$
	ZnCl_2	171°
	$\text{NH}_4\text{Cl} / \text{ZnCl}_2$	173°
Pb = 67% Sn = 33%	Na_2S	$65^\circ 45'$
	Rosin	150°
	NH_4Cl	$157^\circ 30'$
	ZnCl_2	$169^\circ 30'$
	$\text{NH}_4\text{Cl} / \text{ZnCl}_2$	175°

Table III c.
 Contact Angle with Copper
 Surface Emeried

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Na ₂ S	151° 30'
	NH ₄ Cl	159°
	Rosin	167°
	ZnCl ₂	176°
	NH ₄ Cl / ZnCl ₂	177° 30'
Pb = 50% Sn = 50%	Na ₂ S	142°
	Rosin	166° 30'
	NH ₄ Cl	168° 45'
	ZnCl ₂	169°
	NH ₄ Cl / ZnCl ₂	180°
Pb = 67% Sn = 33%	Na ₂ S	149°
	Rosin	163°
	NH ₄ Cl	172° 15'
	NH ₄ Cl / ZnCl ₂	174° 30'
	ZnCl ₂	178°

Table III d.

Contact Angle with Copper

Surface E meried and Etched with KCN

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Rosin	164°
	NH ₄ Cl	164° 15'
	Na ₂ S	168° 30'
	NH ₄ Cl / ZnCl ₂	177°
	ZnCl ₂	178° 30'
Pb = 50% Sn = 50%	Rosin	167°
	Na ₂ S	167° 30'
	NH ₄ Cl	169°
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°
Pb = 67% Sn = 33%	Rosin	166° 30'
	NH ₄ Cl	167°
	Na ₂ S	170° 45'
	ZnCl ₂	174° 30'
	NH ₄ Cl / Zncl ₂	174° 45'

Table IV a.
 Contact Angles with Zinc
 Oxidized Surface

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Rosin	166° 45'
	Na ₂ S	171°
	NH ₄ Cl	173° 15'
	ZnCl ₂	175° 30'
	NH ₄ Cl / ZnCl ₂	180°
Pb = 50% Sn = 50%	Na ₂ S	158°
	NH ₄ Cl / ZnCl ₂	169°
	NH ₄ Cl	171°
	ZnCl ₂	171°
	Rosin	171° 30'
Pb = 67% Sn = 33%	Na ₂ S	0°
	NH ₄ Cl	170°
	ZnCl ₂	171° 30'
	Rosin	172° 30'
	NH ₄ Cl / ZnCl ₂	180°

Table IV b.
 Contact Angles with Zinc
 Surfact Emeried

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Na ₂ S	0°
	Rosin	129° 45'
	NH ₄ Cl	163° 30'
	ZnCl ₂	175° 15'
	NH ₄ Cl / ZnCl ₂	180°
Pb = 50% Sn = 50%	Rosin	141° 30'
	NH ₄ Cl	142°
	Na ₂ S	144°
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°
Pb = 67% Sn = 33%	Na ₂ S	0°
	NH ₄ Cl	118° 30'
	Rosin	160°
	ZnCl ₂	173° 30'
	NH ₄ Cl / ZnCl ₂	180°

Table IV c.

Contact Angles with Zinc

Surface Emeried and Etched with 1 - 1 HCl

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Na ₂ S	0°
	ZnCl ₂	167°
	Rosin	173° 45'
	NH ₄ Cl	174°
	NH ₄ Cl / ZnCl ₂	180°
Pb = 50% Sn = 50%	Na ₂ S	0°
	ZnCl ₂	176°
	Rosin	176° 30'
	NH ₄ Cl / ZnCl ₂	179°
	NH ₄ Cl	180°
Pb = 67% Sn = 33%	Na ₂ S	0°
	Rosin	172° 30'
	ZnCl ₂	176°
	NH ₄ Cl	179° 30'
	NH ₄ Cl / ZnCl ₂	180°

Table V a.
 Contact Angles with Nickel
 Surfact Oxidized

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	NH ₄ Cl	141° 30'
	Na ₂ S	151° 15'
	NH ₄ Cl / ZnCl ₂	153° 15'
	ZnCl ₂	160°
	Rosin	163° 30'
Pb = 50% Sn = 50%	Na ₂ S	122°
	Rosin	138° 45'
	NH ₄ Cl / ZnCl ₂	159°
	ZnCl ₂	171° 30'
	NH ₄ Cl	172° 30'
Pb = 67% Sn = 33%	Rosin	138° 30'
	Na ₂ S	145° 30'
	NH ₄ Cl / ZnCl ₂	149° 30'
	ZnCl ₂	157° 15'
	NH ₄ Cl	167° 30'

Table V b.
 Contact Angles with Nickel
 Surfaced Emeried

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Rosin	68°
	Na ₂ S	143°
	NH ₄ Cl	160°
	NH ₄ Cl / ZnCl ₂	165° 15'
	ZnCl ₂	172° 45'
Pb = 50% Sn = 50%	Na ₂ S	156°
	Rosin	158° 45'
	NH ₄ Cl / ZnCl ₂	160°
	NH ₄ Cl	164° 45'
	ZnCl ₂	180°
Pb = 67% Sn = 33%	Na ₂ S	160° 30'
	Rosin	160° 30'
	NH ₄ Cl / ZnCl ₂	174°
	NH ₄ Cl	175°
	ZnCl ₂	180°

Table V c.

Contact Angles with Nickel

Surface Emeried and Etched with 1-1 HNO₃

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Rosin	132° 45'
	Na ₂ S	156° 15'
	NH ₄ Cl / ZnCl ₂	158°
	NH ₄ Cl	162°
	ZnCl ₂	180°
Pb = 50% Sn = 50%	Na ₂ S	121° 45'
	Rosin	150° 30'
	NH ₄ Cl	162° 45'
	ZnCl ₂	163°
	NH ₄ Cl / ZnCl ₂	168° 30'
Pb = 67% Sn = 33%	Na ₂ S	125°
	Rosin	159° 30'
	NH ₄ Cl	164° 30'
	NH ₄ Cl / ZnCl ₂	176°
	ZnCl ₂	180°

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Table VI a.
 Contact Angles with Tin
 Surface Oxidized

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Rosin	164° 45'
	Na ₂ S	173°
	NH ₄ Cl	177° 30'
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°
Pb = 50% Sn = 50%	NH ₄ Cl	169° 45'
	Rosin	172° 30'
	Na ₂ S	174° 15'
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°
Pb = 67% Sn = 33%	Rosin	154° 30'
	NH ₄ Cl	165° 15'
	Na ₂ S	174° 15'
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°

Table VI b.
 Contact Angles with Tin
 Surface Emeried

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	ZnCl ₂	160°
	Na ₂ S	174°
	Rosin	175° 30'
	NH ₄ Cl / ZnCl ₂	180°
	NH ₄ Cl	180°
Pb = 50% Sn = 50%	NH ₄ Cl / ZnCl ₂	170°
	Rosin	175° 30'
	Na ₂ S	176°
	NH ₄ Cl	180°
	ZnCl ₂	180°
Pb = 67% Sn = 33%	NH ₄ Cl	160° 15'
	Na ₂ S	167°
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°
	Rosin	180°

Table VII a.
 Contact Angles with De-tinned Can
 Surface Oxidized

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Na ₂ S	0°
	Rosin	152° 45'
	NH ₄ Cl	158°
	NH ₄ Cl / ZnCl ₂	169° 30'
	ZnCl ₂	171°
Pb = 50% Sn = 50%	Na ₂ S	0°
	Rosin	155° 30'
	NH ₄ Cl	163°
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°
Pb = 67% Sn = 33%	Na ₂ S	51°
	Rosin	155° 45'
	NH ₄ Cl	162°
	ZnCl ₂	174°
	NH ₄ Cl / ZnCl ₂	175°

Table VII b.
 Contact Angles with De-tinned Can
 Surface Emeried

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Na ₂ S	0°
	NH ₄ Cl	138°
	Rosin	154°
	NH ₄ Cl / ZnCl ₂	160°
	ZnCl ₂	170°
Pb = 50% Sn = 50%	Na ₂ S	96°
	Rosin	165°
	NH ₄ Cl	180°
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°
Pb = 67% Sn = 33%	Na ₂ S	0°
	NH ₄ Cl	160°
	Rosin	161° 45'
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°

Table VII c.

Contact Angles with De-tinned Can

Surface E Meried and Etched with Picric Acid

Solder Used	Flux Used	Average Contact Angle
Pb = 33% Sn = 67%	Na ₂ S	116°
	Rosin	153° 30'
	NH ₄ Cl	167° 30'
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°
Pb = 50% Sn = 50%	Na ₂ S	55°
	Rosin	142° 30'
	NH ₄ Cl	180°
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°
Pb = 67% Sn = 33%	Rosin	143° 30'
	NH ₄ Cl	162° 30'
	Na ₂ S	163°
	ZnCl ₂	180°
	NH ₄ Cl / ZnCl ₂	180°

DISCUSSION

The results obtained with the ammonium chloride, zinc chloride, the mixture of equal parts of the two chlorides, and rosin were similar to those obtained in the preliminary experiments. With the plate metals used, the zinc chloride alone or mixture of the two chlorides proved to be the best flux.

The results obtained when sodium sulphide was used as a flux have not proved satisfactory. The temperature required is higher than that obtained by use of the Bunsen burner. The sodium sulphide apparently reacts with the plate metal, forming the sulphide. The heat required to reduce the sulphide could not be obtained with the Bunsen burner alone. With the aid of a blowpipe wetting could be effected.

The field possible for exploration has barely been touched by the material in this paper, and it is the author's opinion that this field should be further investigated. Innumerable possibilities present themselves by varying the solders, fluxes, and conditions under which the experimentation is carried on.

SUMMARY

1. Rosin will protect a surface from oxidation and will clean solder, but will not always clean an oxidized surface.

2. The inorganic salts, ammonium chloride, zinc chloride, and a mixture of the two chlorides clean an oxidized surface as well as protecting a previously cleaned one.

3. The common soft-solder fluxes will not cause wetting of the aluminum. This is probably due to the resistant aluminum oxide film which forms and is not broken down by the fluxes.

4. Best results are obtained when the plate metal is previously cleaned and the flux and solder placed on the metal before the latter is heated.

5. Sodium sulphide has not proved satisfactory as a flux with the conditions maintained during the experiments.

ACKNOWLEDGEMENTS

To Dr. Curtis L. Wilson and Dr. Ettore A. Peretti of the Department of Metallurgy of the Montana School of Mines, under whose able guidance this work was done, my obligation is herewith gratefully acknowledged.