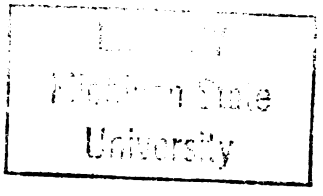

This is to certify that the

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THE EFFECTS OF IMPURITIES AND THEIR REMOVAL FROM NICKEL PLATING SOLUTIONS

By

William Damude Gordon

A THESIS

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This research was undertaken at the request of, and under a grant from, the American Electroplater's Society.

The material in this thesis consists of two separate portions of research. An extensive and comprehensive search was made of the literature. The bibliography at the end of this thesis is considered to be a complete listing of references on the effects of impurities in nickel plating solutions, and their removal. This bibliography has been arranged in numerical order by years starting with 1901. Within each year the articles are listed alphabetically according to the authors' name.

The first portion of this thesis consists of a critical review and compilation of abstracts of the literature covered in the bibliography. This work was designed to bring together in one paper the essential facts which have been brought out in the literature on this subject, and to point out where further information can be found on any particular impurity. A critical discussion and evaluation of the various papers, in view of limited accurate experimental data and experience, is given.

ALUMINUM

The Effects of Aluminum in Nickel Plating Solutions: The first case of the presence of aluminum in nickel plating solutions seems to have been reported by Watts (236) in 1927, when he reported that the addition of the hexahydrate of aluminum chloride increased the throwing power of the bath so remarkably that it approached that of a cyanide copper bath. Citric acid was also added to keep the aluminum in solution as a complex. In 1935, Harr (278), however, reported that aluminum sulfate led to a bad effect on metal distribution in both high and low pH solutions. Harr also found that a low concentration of aluminum sulfate decreases, while a large concentration increases, the critical current density. He also reported a dark, burned deposit from solutions containing aluminum, which he attributed to the precipitation and codeposition of aluminum hydroxide.

Dark deposits were also found by O'Sullivan (283) who reported, "Aluminum can accumulate in the cathode film until colloidal compounds are formed, and if this process proceeds far enough the structure of the deposit is profoundly altered, becoming black or "burnt." Since these black deposits have been found to contain appreciable amounts of aluminum, whereas none could be detected in white deposits, the supposition that the change is due to the co-precipitation of colloidal aluminum compounds with the nickel seems justified."

Puri and Juneja (570) added an aluminum hydroxide sol to a nickel plating solution and reported improved luster, but also increased hardness, and brittleness. Martin (657) recently found that aluminum

when present in a nickel plating solution, causes increase stress at a pH of 5.5; but since aluminum hydroxide precipitates at a pH of 4.0, it is more likely that the stress is caused more by the colloidal aluminum hydroxide rather than an effect of the aluminum ion itself.

A trace of aluminum sulfate has been reported as an excellent brightening agent, and here again the effect is attributed to the capacity of the aluminum to pass into the colloidal state by hydrolysis. (615)

In general it can be assumed that aluminum is deleterious to a nickel plating solution, and that its presence in other than trace amounts causes dark, "burnt", deposits with a tendency toward stress and brittleness.

The Removal of Aluminum from Nickel Plating Solutions: Since Aluminum hydroxide readily precipitates at a pH of 4.0 or above, it is readily separable from a solution containing no citrates, tartrates, etc., which might form soluble complexes with aluminum. The pH is raised by adding either nickel hydroxide or nickel carbonate (370) or by the addition of magnesium and calcium carbonates (349), or by aeration with one of these methods. The precipitate which forms is gelatinous, and may not settle to the bottom of the tank, so that filtration using a suitable filter aid is the most effective filtration procedure.

THE AMMONIUM, POTASSIUM AND SODIUM IONS

The Effects of Ammonium, Potassium, and Sodium Ions in Nickel Plating Solutions: The ammonium, potassium and sodium ions have been common ingredients of nickel plating solutions practically from the beginning. With the popularity of the double salt solution in the early days of the industry, these ions were not considered as impurities but rather as essentials to the bath. The chlorides of sodium and ammonium were common conducting salt additions. The first Quarterly Review of the American Electroplater's Society carried an article by W.S. Barrows (46), in which he stated that the addition of the chlorides of ammonium and sodium increases electrical conductivity, and retards the formation or deposition of hydrogen at the cathode. He also states that sodium chloride usually causes turbidness of the solution, and may result in porous deposits. The leaders of controlled electroplating in this country and abroad are divided on which salts of sodium and ammonium produced the desired results. Sizelove (88), Haas (94), Merrick (148), Hammond (178), Faint (216), Underwood (262), and Krause (328) advocated the use of ammonium chloride as a conducting salt, and claimed superior results on the deposit. Brown (93) and an anonymous German author (195) favored the addition of ammonium sulfate. It is quite likely that the favorable results of the ammonium chloride were due in a large part to the chloride anion, and the better anode corrosion, etc. which are among the effects attributed to the chloride ion.

Advocates of sodium chloride additions were Neil (96), Proctor (124),

and Foss (252), the latter two authors favoring this addition along with citric acid in the plating of sheet zinc and aluminum. Werner (624) preferred the use of sodium citrate, and ammonium chloride for plating zinc. Two Russian authors, Leusorov (486) and Kochergin (520), claim the preference for sodium chloride, the former for plating printer's type and the latter for producing detachable films of nickel.

There was also a group of men who considered sodium sulfate as an excellent conducting salt addition. These included Matuschek (118), Gordon (174), and Ballay (342). With this wide diversity of opinion there would probably be at least one person who would take an intermediate position. Dorrance and Gardiner (241), in an article on polarization and resistivity in nickel plating solutions, found no appreciable difference between potassium, sodium, ammonium, or nickel ions in their conductance effect when combined with chloride anions. Thompson (212) advocated use of a large amount of sodium sulfate and a small amount of ammonium chloride in plating zinc and zinc-base die-castings, while Lemens and Dupont (524) showed no preference for either ammonium chloride or sodium chloride in plating cast iron.

The presence of these ions in solution has been given the credit for almost any plating effect known. Pitschner (284),(406) wrote a pair of articles concerning the buffer action of ammonium chloride in nickel plating solutions. Hirsch (350) reported that the addition of ammonium hydroxide (24-60 g/l.) made the nickel deposit brighter in barrel nickel plating of fabricated sheet zinc parts. Haring (179) in his classic work on throwing power reported that sodium chloride increased throwing power more than the addition of ammonium chloride. Ammonium sulfate and sodium fluoride, however, were found to reduce

throwing power. Mothersall (332), in a study of pitting, reported that ammonium, sodium, and potassium ions have a stabilizing effect in preventing flocculation of the basic colloids precipitated at the cathode face. Without these ions the solution produced badly pitted deposits. On the matter of anode corrosion, the use of ammonium and sodium chlorides has been reported as increasing anode corrosion (114) and (99); but here the corrosion effect was not due to the alkali ions but to the chloride anions. Thomas and Blum (189) proved this by showing that ammonium sulfate alkali or fluorides did not materially increase corrosion. Liscomb (79) was set against the addition of ammonium hydroxide to increase anode corrosion, pointing out that ammonium sulfate is formed, and in sufficient concentration double nickel salts would form with resulting loss of efficiency. Bennett, Kenny, and Dugliss (59) early came forward with the theory that the addition of ammonium hydroxide increases efficiency and gives good deposits. The improved efficiency is attributed to the neutralization of hydrogen ions as the hydrogen forms at the cathode. In line with these electrochemical effects, Watts (639) recently reported that in plating from a nickel sulfate bath of low concentration (10 to 40 g./l.), to which had been added large amounts of sodium sulfate (200-250 g./l.), no plate deposited on the front of the cathode but did on the back. He attributes this phenomenon to the formation of an alkali film on the front of the cathode.

The alkaline cathode film reported by Watts may account, in part, for the increased hardness associated with nickel plating from solutions containing these alkali ions. Macnaughton, Gardam, and Hammond, (332) in examining hard nickel deposits, found them to contain basic

colloidal matter which is of a positive charge. Snelling and Thews (613) also reported that the hard deposits from a nickel bath containing ammonium and chloride ions in solution, also contain these ions in the plate. Blum and Kasper (366) found that ammonium sulfate increases hardness whereas sodium sulfate did not materially increase hardness. They also found that ammonium chloride increases hardness whereas calcium chloride did not have such an effect in a nickel chloride bath. Wesley (497) also advocated hard nickel solutions containing ammonium sulfate and potassium chloride, and ammonium chloride and potasssium chloride, and Weisberg, (542) made a similar report on ammonium sulfate additions. Wesley and Roehl (640) also reported a hard nickel bath containing ammonium chloride alone. Makar'eva (482) found generally that the addition of potassium, ammonium, and magnesium ions to sulfate nickel baths gave deposits more lustrous, harder, and more uniform in texture. He also reported that baths containing potassium chloride and fluoride produced less porous deposits.

In regard to the effects of these ions on the appearance of the plate, Moline (648), Poor (652), and Schore (653) felt that ammonium sulfate is a very important constituent in black nickel baths where a jet-black color is desired. In an early experiment in their laboratories, the old Brass World reported (29) that additions of ammonia to nickel plating baths produced deposits having a dark, iron-like color, and lacking tenacity and toughness, and were hard and brittle. Nelson, (108) also claimed that the additions of sodium or ammonium chloride to a double salt solution produced a dull plate, and Under-

wood (467) reported that the addition of sodium chloride may cause brown-stained work.

Reviewing these varied effects, several general statements can be made as fairly acceptable facts: first, that the addition of these ions causes a harder, more brittle deposit to form; second, that their use in salt form as conducting salts does not produce results to warrant their use, and that the other effects produced outweigh any advantage they have in increasing conductance; third, that the use of the chlorides and sulfates in plating on zinc and its alloys, aluminum, and cast iron is probably of some value.

The Removal of Ammonium, Potassium, and Sodium Ions from Nickel Plating Solutions:, The only apparent method for removing these ions from solutions is by filtration through materials having zeolitic properties. (664)

ARSENIC

The Effects of Arsenic in Nickel plating Solutions: Arsenic in nickel-plating solutions is present primarily as an ingredient in black nickel baths. Slater (24), Watts (54), Allen (104), Blum (105), and Barrows (224) published formulas for black nickel baths containing arsenic. The arsenic was generally added as "white" arsenic or arsenic trioxide. Hogaboom, Slattery, and Ham (117), made a study of black nickel baths at the Bureau of Standards, and investigated the deposits formed; but they did not report the presence of arsenic in the deposits. Keama, (171) however, reported that the nickel deposit formed from a black nickel solution is a nickel-arsenic alloy.

The use of arsenic as a brightener was reported by Mazucha (95) who also reported that the resulting plate was not only brighter, but smoother. Diggin (664) reported that arsenic in small concentrations has a brightening effect, but that the deposits are brittle. He also pointed out the very interesting fact that these deposits when heated, as in soldering, will turn black. Wittum (544), and Raub and Wittum (534) found that arsenic causes dull, streaked, and brittle deposits, and observed the generation of arsine at the cathode. Morokov, Stepanov, and Moroko (567) found that arsenic trioxide additions promoted hydrogen adsorption and codeposition of arsenic. The tensile strength of the deposit was also decreased. They consider the maximum permissible concentration of arsenic to be 10 mg./l.

To summarize, arsenic can be considered to be detrimental in nickel plating baths. In concentrations above 10 mg./l. dark, brittle, low

tensile strength deposits may result.

The Removal of Arsenic from Nickel Plating Solutions: The Liscomb method of purification has been found (664) to be successful in removing arsenic. This treatment consists of adding about twelve ounces per gal. of ferrous sulfate to the bath, then raising the pH of the bath to 6.2 to 6.4 with nickel carbonate or nickel hydroxide, and subsequently oxidizing with hydrogen peroxide at 140 degrees Fahrenheit. A large mass of gelatinous ferric hydroxide forms, which on settling out carries much contaminating material with it. After settling, the bath is preferably filtered, or the clear solution decanted off. An article has also been written by Skowronski (261) on the refining production of nickel salts used for electroplating, and mention is made of procedure for removing arsenic, copper, iron and zinc.

BORON

The Effects of Boron in Nickel Plating Solutions: The presence of boron in nickel plating baths is almost exclusively as boric acid. Since boric acid is now considered as an essential ingredient in nickel baths, it can hardly be considered as an impurity. No attempt will be made, therefore, to present a comprehensive list of all the references which deal with the presence of boric acid in the solution. However, a few select references will be discussed which will give a general viewpoint of the various effects which have been observed by various investigators.

Boric acid in nickel baths is present primarily for its buffering properties. It prevents the solution from becoming too alkaline during electrolysis, which may result in the formation of nickel hydroxide and result in rough, dark deposits. (179) The first use of boric acid, however, appears to have been primarily from the viewpoint that it prevented pitting and produced a soft white deposit. (32). A large amount of boric acid in the bath was advocated with this end in mind. Besides the decreased tendency for the bath to pit, and the whiter deposit produced, the deposit was also claimed to be softer. (31). Liscomb (80) and Proctor (286) felt that the important effect was to prevent pitting, whereas Sizelove (38) mentioned primarily the whiter, tougher, and more lustrous plate produced. An article by the Chicago Branch of the American Electroplater's Society (114) also recommended the addition of boric acid to produce a white background. MacNaughton (286) felt that the presence of boric acid was an absolute

necessity to producing a soft nickel deposit from a bath of pH 2.0 to 6.0. The absence of boric acid, he felt, led to hard, cracked, and pitted deposits. In baths of pH 7 to 9 Ballay (341) found that boric acid caused premature hydroxide precipitation. The deposits were, however, unsatisfactory at this pH range, with or without the presence of boric acid. At this pH range boric acid is obviously not a satisfactory buffering agent.

The use of boric acid with hydrofluoric acid has appeared periodically in the literature for over thirty years. Hollard (49) was probably the first to use this combination claiming it as a means for preventing liberation of hydrogen at the cathode. After a number of years, Lovering (123) provoked new interest in fluoborate additions with his "magic fluid." He claimed better anode corrosion with hydrofluoric acid or fluoborate additions, and also claimed that anode sludge formation was prevented, and cathode efficiency increased.

The use of sodium perborate has been mentioned also, primarily as an oxidizing agent in overcoming pitting due to organic contamination. Proctor (206) was one of the first to use this compound, and his claim for it was that it prevented the formation of hydrogen blanket at the cathode, and thus prevented resulting brittleness and pitting. Sodium perborate was also added by Underwood (221) to a solution which yielded brittle and pitted deposits and the condition was corrected.

It is interesting to note that Fink and Rohrman (296), in their experiments on the preparation of pure electrolytic nickel from a Watt's type bath, spectroscopically found cobalt, iron, and copper to be absent,

but did find traces of boron and sodium present in the nickel. It is doubtful that there are ever any more than trace amounts of boron-containing materials ever codeposited with nickel, but this may be a fact which has been long overlooked in studies of nickel deposition.

The Removal of Boron from Nickel Plating Solutions: The removal of boric acid from solution does not appear to be necessary, since, it is known that nickel plating solutions can tolerate amounts of boric acid, limited only by its solubility in the bath.

BROMINE

The Effects of Bromine in Nickel Plating Solutions: The Effect of the bromide ion on polarization and resisitivity in nickel plating solutions was studied by Donance and Gardiner (241) to determine whether this ion is superior to the chloride ion in reducing anode polarization. Both ions considerably reduced anodic polarization, but no advantage for the bromide ion was observed over the chloride ion. Since the bromide ion acts like the chloride ion its presence in a plating solution would probably not be detrimental. Since the bromide salts are more rare and expensive than ~~that~~ the chlorides, it is doubtful if their presence in plating solutions is very likely, other than in trace amounts associated with chlorides.

CADMIUM

The Effects of Cadmium in Nickel Plating Solutions: The first notice of the use of cadmium as a brightener in this country was made by Proctor (68) in 1915. Small sticks of pure cadmium connected to the anode rods were noticed by him in a nickel plating bath in a Connecticut company. On questioning the foreman he found that this procedure had been recommended to him by an Englishman. The use of cadmium in this manner had evidently been used in England prior to this date with good results. Proctor's paper immediately caused much interest in the use of this metal as a brightener. Articles by Mills (100) and Carlson (113) followed a few years later, which recommended the addition of cadmium as cadmium chloride, which produced excellent, bright, smooth deposits. By 1925, the effect of cadmium in nickel plating solutions was quite well known, and Proctor (170) followed up his original paper with one in which he stated that the rise of cadmium metal as small anodes along with the nickel anodes is not to be recommended; but he strongly advocated the addition of cadmium in salt form, preferably as the chloride. He also pointed out the dangers of using too much cadmium as causing brittle deposits streaked with dull gray marks, and deposits which tend to be tinted yellow on drying. Since that time numerous authors have published articles on the use of baths containing cadmium as a brightener-Proctor and Sizelove (207) Underwood (262) and (263), Raub and Bildmaier (392), Vozdvizhenskii and Suleimanova (435), Young (592), and Pollack (636). Some excellent work has been done on the limits of cadmium which a solution is able to tolerate and still produce a satisfactory plate. White and Proctor

(227) found that the brightening effect of cadmium chloride increased with concentration up to 0.05% in a standard still bath. From 0.05 to 0.10% there was no apparent change in the deposit. Above 0.10% the deposits become increasingly poorer in quality. Evlannikov and Neiman (506) pointed out that cadmium in excess of 0.5g./l. in a nickel bath causes hard cracking deposits. Wittum (544) considered the upper limit to be 1.6%, which is quite a bit higher than the 0.10% maximum of White and Proctor. Evidently the bath compositions or the current densities used are different and Wittum's bath can tolerate more cadmium under these conditions. Meyer (607) considered 0.013 to 0.067 oz./gal. as a favorable range, and 0.13 oz./gal. as causing poorly adherent and black deposits up to 6 amp./sq. ft., and bright and spotted from 6 to 22 amps./sq.ft., and clouded and streaked above, 22 amps./sq.ft.. He found that concentrations above 0.67 oz./gal. caused the deposits to be brittle and non-adherent. Raub and Wittum published a pair of articles on the use of cadmium (534), (572) in which they considered cadmium an unsuitable brightener since it has a detrimental effect on the deposit. They attributed the bands formed in most bright nickel deposits to the codeposition of foreign matter in a periodically changing sequence. Cadmium is one of the metals which they considered as depositing as alloys with nickel in this manner. They also pointed out that since cadmium deposits with less polarization than nickel, the amount of cadmium in the deposit will be of higher concentration than the cadmium in the solution.

Haring (180), Hoefer (289), and Sizelove (462), all reported that the presence of cadmium in a nickel plating solution decreased throwing power. Haring also found that deposits from solutions containing 0.001N cadmium were very spongy, alternately bright and gray except at high current densities, where a satisfactory, and brighter than normal plate is obtained. Hoefer also found the presence of cadmium reduced bath voltage, while Sizelove reported that the deposit from a cadmium containing solution has a tendency to stain; but if cobalt is used with the cadmium as a co-brightener, the color is greatly improved. Eckelmann, (347) however, found that cadmium tends to promote the codeposition of nickel hydroxide. This theory was also shared by an anonymous German author (365) who attributed the brightening action of cadmium to the fact that it forms basic colloidal salts near the cathode which are codeposited with the nickel, resulting not only in a bright plate, but a hard and brittle one as well. An article has also been published by Lapin and Matveeu (419) on the effect of cadmium salts in the nickel plating solutions on the properties of the deposit. Further data could not be obtained on the article, but it probably contains some interesting information.

The Removal of Cadmium from Nickel Plating Solutions: Pollack (491) discovered that as plating baths become older, they become less susceptible to the influence of impurities. He corrected striped deposits caused by excess cadmium and zinc by an artificial aging process. Wiesner (655) did some excellent work on the effectiveness of the hy-

droxide precipitation method of purification. He found, in a solution buffered with boric acid, that with a cadmium content of 0.5 g./l., no precipitation occurred up to a pH of 7.1 upon addition of ammonium hydroxide. Since 0.5 g./l. cd is well above the permissable limit, the use of this method is not effective. Between a pH of 6.2 and 6.3 Wiesner found that the solution became turbid and above 6.3 a large quantity of nickel hydroxide precipitated. The nickel hydroxide also carried out some cadmium, but the amount was too small to warrant the loss of the nickel.

The only really satisfactory method of removing cadmium appears to be selective electrolysis on dummy cathodes at 2-3 amps./sq.ft..

CALCIUM

The Effects of Calcium in Nickel Plating Solutions: In 1916, B.E. Miller (83), a practical electroplater, published a paper in the "Monthly Review", in which he strongly advocated the use of calcium chloride as a conducting salt. He noticed that a scum was formed upon adding the salt and allowing the solution to stand stagnant overnight. The next morning he would scrape the scum off and discard it and he claimed the resulting nickel deposit from this solution was soft and malleable. The scum fascinated him, and he challenged readers with scientific background to explain this formation. In the next issue of the "Review", Robins (86) explained that the action of the calcium chloride was to precipitate as calcium sulfate on being added to the bath, replacing the sulfate ions with chloride ions. Since commercial calcium chloride, at this time, was known to contain about 16% magnesium chloride, this compound was attributed with forming the sludge. To more directly accomplish the same end, Robins advised the use of nickel chloride. This one instance appears to be the solitary case of an intentional addition of calcium to a nickel plating bath. Twenty-three years later calcium again came into notice with an article by Meyer (429) which pointed out that rough deposits could be caused by suspended calcium sulfate in the solution coming from hard water. Hogaboom (515) quickly followed with an article pointing out that dull, pitted, rough, stained deposits could result from the use of hard water in making up and replenishing solutions. Diggin, (596) also followed with an investigation of the effect of the various water impurities on nickel deposits. He found that calcium

of itself does not seem to affect the deposit, but that calcium sulfate forms, and can cause bright nickel to plate dull and spotted, generally on horizontal surfaces. On microscopic examination of the spots, it was found that they were actually pits, thereby reducing the corrosion resistance of the deposit. Kushner (629) also found that calcium, introduced into a plating solution by drag-in of rinse water, caused bright nickel in some cases, to plate dull. Diggin (664) recently pointed out that calcium can exist in solutions up to the point of saturation without causing harm, the chief trouble arising when it precipitates as calcium sulfate causing rough and spotted deposits.

The Removal of Calcium from Nickel Plating Solutions: The best solution to the presence of calcium in nickel plating baths is to prevent it's admission to the bath by using only soft or deionized water. However if it is present in the bath, a simple filtration will remove the precipitated calcium sulfate.

CARBON

The Effects of Carbon in Nickel Plating Solutions: The effects of carbon discussed here will be only those attributed to the element itself, or to its oxides, and inorganic compounds. The great mass of organic materials which have been reported will be treated later in a section on organic matter.

Madsen (182), reported in 1924 the presence of carbon in nickel electrodeposits from an apparently inorganic solution. Madsen (188) also found 0.02% carbon in electrolytic nickel deposits when anodes were used containing 0.20% carbon. Lambris (26) came forward earlier in 1910 with four conclusions as to the cause of the presence of carbon in electrodeposits, his experiments being scientifically done by purposely adding carbon monoxide, carbon dioxide and acetylene to the solution. His conclusions were:

(1) The absorption of carbon by the nickel was entirely due to a gas reaction.

(2) The gases which will introduce carbon into the nickel are carbon monoxide, carbon dioxide, and acetylene.

(3) Oxalic acid is partly reduced to acetylene upon nickel cathodes.

(4) The carbon in electrolytic nickel is present in the form of carbide.

Kohlschütter and Nägeli (127) later found that the carbon is not deposited visibly upon the film, but in the interior of the metal, and that the allotropic of carbon in these deposits is graphite. Frölich

(177) confirmed the work of Lambris, finding also that the two oxides of carbon and acetylene contaminated the deposit. He also found that polarization of nickel in solutions containing organic matter may cause contamination of the metal. The organic impurities thus introduced are situated in the interior of the solid metal.

Carbonates have been an ingredient of black nickel solutions, (24), (104), (105), but the exact effect does not seem to be known. More recently Anderson (401) found that the presence of carbon monoxide, carbon dioxide, and carbonates have an embrittling effect on the deposit, leading to edge cracking. Spinger, (465) considered the addition of carbonates essential to nickel plating with insoluble anodes, but also admitted that rough deposits might result. Weisberg (582) found that a solution which has been accidently contaminated with activated carbon from a filtering operation will lead to rough deposits, and once in the solution, it is difficult to remove by filtration.

Practically all bright nickel baths which contain organic brighteners will produce carbon contaminated deposits. Pinner, Soderberg, and Baker (609) reported that certain bright nickel deposits contained 0.05 to 0.2% carbon.

The Removal of Carbon from Nickel Plating Solutions: Since the source of carbon in nickel deposits is essentially organic matter, the removal of these compounds is discussed in that section.

CHLORINE

The Effects of Chlorine in Nickel Plating Solutions: Chlorine, or rather the chloride ion, is hardly considered as an impurity in nickel plating solutions. Its presence is generally considered as essential to good anode corrosion, especially in using the 97-99% pure anodes of today. It is interesting to note however, that there are many effects attributed to chlorine and the chloride ion, which should be mentioned.

Blassett, (33) early advocated the use of chloride additions to the nickel plating bath to prevent the formation of hydrogen bubbles on the cathode with resultant pitting. Although chlorides were known to be compatible with nickel solutions, Haas, (94) reported that the addition of hydrochloric acid to a bath caused dark deposits which were also brittle. Since sulfuric and nitric acids produced similar results, the effect is undoubtedly due more to the acid properties of the addition rather than an effect of the chloride ion. MacNaughton and Hammond (203), MacNaughton, Gordon, and Hammond (322), and MacNaughton (386) have come forward with the theory that chlorides seem to aid flocculation of the basic compounds of nickel, and therefore increase pitting. The joint presence of the chloride ion and alkali ions, however, reduces the flocculating tendency, and thus do not stabilize the gas bubbles forming on the cathode. Borrance and Gardiner (241) have made the observation that the chloride ion considerably reduced anodic polarization. Schlotter, in two articles (395) (396), showed that the chloride ion and other anions can exist in coherent cathodic deposits. They may take part in cathodic deposition and also enter into the crystal lattice of the metals, thus changing

the lattice constant. This observation was affirmed by Shelling and Thews, (613) who found the presence of chloride ions in hard nickel deposits plated from a bath containing chloride. There have been many advocates of the high chloride nickel plating baths in the past few years, but Fedtieff and Kindred, (275) have revealed a unique argument for the use of nickel chloride solutions instead of the customary chloride-sulfate baths. They found that electrolysis from a chloride solution avioded the possibility of sulfur contamination, particularly in electrowinning nickel.

CHROMIUM

The Effects of Chromium in Nickel Plating Solutions: The first investigation of the effects of chromium in nickel sulfate solutions appears to be that of Russo (39), who in studying the anodic behavior of nickel in the presence of chromous salts found that it hindered, but did not entirely prevent the passivation of nickel, and that it modifies the polarization values slightly. Hallard (41) reported the following year that spongy deposits of nickel caused by hydrogen formation could be prevented by addition of small amounts of sodium dichromate to the bath. MacNaughton and Hammond (280) were the first to emphasize that the possibility of chromium contamination of nickel plating solutions is probably frequent in plants doing both chromium and nickel plating. They undertook an exhaustive study of the effects of bath chromic acid and chromium sulfate, and found that amounts of chromic acid from .0248 g./l. to 0.20 g./l. caused decreased cathode efficiency, and at 0.22 g./l. the deposition closed completely. As the concentration of chromic acid increased, gassing at the cathode also increased. The presence of from 0.01 to 0.025 g./l. of chromic acid was found to cause a considerable increase in the stress of the deposits, and many of the plates exfoliated in the bath. In adding chromic sulfate in concentration of chromium content of 0.149 g./l., the cathode efficiency steadily fell to 81%. The deposits also increased in brightness as concentration increased, but with increasing tendency to peel. When enough chromic sulfate was added to be equivalent to that concentration of chromic acid which stopped deposition completely, the anode efficiency was not noticeably affected.

Hothersall and Hammond (381) continued the study of the effect of chromic acid, and, besides confirming the results of Macnaughton and Hammond (280), they found that an appreciable content of chromic acid increases the evolution of hydrogen, and does not stop pitting. With a given concentration, the effect of chromic acid decreasing the cathode efficiency and causing strained deposits, was more accentuated in low pH solutions than in higher ones. They suggested that chromic acid functions in two ways in a nickel plating bath:

(1) "With low concentrations, initial depolarization results in precipitation of basic nickel compounds, containing chromium, at the cathode face, the nickel ion concentration being thereby depleted. Hydrogen ion discharge and reduced cathode efficiency are thus favored."

(2) "With higher concentrations, chromic acid interacts chemically with the cathode metal with the formation of a selectively permeable film on its surface; nickel deposition is completely inhibited as a result. With intermediate concentrations both of these affects may occur simultaneously."

Pietrafesa, (336) also reported that the presence of chromium in a nickel plating bath lowered the cathode efficiency and yielded poor deposits. He attributed the cause to the diminuation of hydrogen overvoltage by formation of chromium chromate and chromic hydroxide, which in a colloidal state are deposited by cataphoresis on the cathode. Puri and Seth (612) also investigated the action of chromium sols in the electrodeposition of nickel on copper, and observed that there was a tendency for the metallic sol to increase the luster of the

nickel deposit, but that the oxide sols rendered the deposit very hard and uniform. Martin (657), in his study of the causes of stress in nickel deposits, found that in a low pH Watt's type bath hexavalent chromium had little effect on stress. At a pH of 5.5, however, hexavalent chromium increases stress. Since this is in agreement with the findings of Macnaughton and co-workers, it appears that the stress effect is due more to the coprecipitation and colloidal effect of chromic hydroxide rather than to metallic chromium itself, since a sol of metallic chromium had no detrimental effect. Skalozubov, and Vlasova (579) have found that an alloy of chromium and nickel can be deposited from a nickel sulfate-boric acid solution to which has been added large amounts of chromic acid. With other conditions constant the chromium content of the alloy increases with current density. The alloy composition can also be varied by varying the bath composition. Similar results were found by Skalozubov and Goncharova (578), who deposited nickel-chromium-iron alloys from sulfate solutions of nickel and iron to which had been added boric acid and chromic acid. From this it would appear that satisfactory deposits can be obtained if large amounts of chromic acid are present, but that no deposition at all will occur at very low concentrations. Solanki and Singh (615) have reported that trace amounts of chromic acid will give rise to powdered deposits of nickel or iron.

In general, therefore, chromium contamination of nickel solutions leads to reduced cathode efficiency, and even total inhibition of deposition. The deposits tend to be spotted and highly stressed.

The Removal of Chromium from Nickel Plating Solutions: Several methods of removing chromium contamination have been published. MacNaughton and Hammond (290) not only pioneered the first investigation of the effects of chromium contamination of nickel solutions, but were the first to offer a practical form of removal. They recommended two procedures, one from a boiling solution as follows: "Ferrous sulfate is added in slight excess of the amount required to reduce the chromium present to the trivalent form. Powdered nickel carbonate--is added to the solution in amount adequate to ensure that on boiling, the pH will be raised sufficiently to cause the complete and rapid hydrolysis of the trivalent chromium salt; usually 2 grams of nickel carbonate per liter is sufficient. Upon boiling for one hour the chromium present in the solution is precipitated as chromic hydroxide, together with ferrous and ferric hydroxides. These substanced together with nickel carbonate are removed by filtration. The pH is then adjusted by the addition of sulfuric acid, and the solution is suitable for use." Another method they proposed used freshly precipitated nickel carbonate added to a nickel sulfate bath, which was maintained at 35°C for about 18 hours, while air was bubbled through the solutions simultaneously. The chromium was completely hydrolyzed, precipitated as hydroxide, and settled out with whatever ferric hydroxide which was also precipitated, and the excess nickel carbonate. Kaub and Bihlmaier (392) noted that chromium is difficult to remove but contend that precipitation as an insoluble chromate is a solution. Smith (538) advises the method of Macnaughton and Hammond, pointing out that not only is chromium removed but also iron, copper, and zinc.

Weisberg (541) pointed out that the Liscomb method will remove chro-

mium contamination, but that it could also be removed by electrolyzing the solution at a pH of 2 with a very high current density. At this pH and current density, no nickel precipitated, and the solution gases heavily. He also stated that trivalent chromium causes as much trouble as chromic acid, and that a satisfactory procedure for removing trivalent chromium is to electrolyze the solution with lead anodes, which oxidizes the chromium and precipitates it as insoluble lead chromate. The solution is filtered to remove the lead chromite, and the excess lead must then be removed with cementation of finely divided nickel or by using Liscomb's purification method. Wiesner (655) has shown that a pH of 7.0 is needed to precipitate out hexavalent chromium very completely by the addition of sodium hydroxide or ammonium hydroxide and keeping the solution at 100°C for three hours. Trivalent chromium is removed in a buffered nickel plating solution at a pH of 5.0-5.2 after heating for 3 hours at 100°C. It is obvious from Wiesner's study that the only practical way to remove chromium by precipitating the hydroxide is to first reduce it to the trivalent state, and then raise the pH to 5 and either boil the solution for several hours, or let it stand cold for over 24 hours. This point has also been made by Waite (666). Pietrafesa (336) in his study of chromium contamination found that the bath could be regenerated by prolonged electrolysis. Diggin (664) recommended treatment of chromium contaminated nickel plating solutions with carefully calculated amounts of lead salts, particularly lead carbonate. Under these conditions highly insoluble lead chromate is formed, and since lead carbonate and lead sulfate are also quite insoluble, there would ordinarily be no trouble from lead contamination as a result of this treatment.

Krause (562) has offered a method of preventing the deposition of the hydroxides of iron, manganese, and chromium during nickel deposition which consists of adding acetic, citric or tartaric acids to the bath. This does not remove the metallic contamination, but it presumably removes, temporarily, at least, the bad effects.

COBALT

The Effects of Cobalt in Nickel Plating Solutions: The use of cobalt in nickel plating solutions dates back to at least 1871, when Nagel obtained a patent for a plating bath containing the double sulfates of cobalt and nickel to deposit a nickel-cobalt alloy.

From this early beginning, there has been a multitude of articles written on the deposition of cobalt-nickel alloys of many compositions. Since the chemistry of cobalt and nickel are so similar, it is difficult to prepare one metal without at least trace contamination by the other. Although the various alloys have physical properties of their own, the amount of cobalt in the solutions from which they were plated, excludes cobalt from consideration as an impurity.

In small amounts, however, cobalt has been reported to have certain effects. Sizelove (462) reported that cobalt used with cadmium as brighteners in a nickel plating bath resulted in a better color of the deposit, since the cobalt neutralizes the yellow shade often resulting from the use of cadmium alone. Meyer (607) has pointed out that 0.13 to 0.65 oz./gal. of cobalt has a slight brightening effect and decreases the ductility but slightly. Schelman (347) has reported that cobalt acts as a brightener, but that it also tends to promote the codeposition of nickel hydrate. It has also been reported by Fink (625) that a 50-50 deposit of cobalt and nickel exhibits better corrosion resistance than either cobalt or nickel deposits alone. The original work on bright deposits of cobalt-nickel alloys done by Fink and Lah (269) reported bright deposits in the range of 25 to 45% cobalt, without any other brightening material. The use

of cobalt with organic brighteners in modern baths, according to Weisberg (583), should produce an alloy of about 18% cobalt for least sensitivity to impurities, and maximum luster.

The Removal of Cobalt from Nickel Plating Solutions: Many separations of cobalt and nickel have been claimed by various authors, but most of these were done on a small basis, such as in electrolytic quantitative analysis; or the method employed is such that if used in an electroplating bath, the bath would be ruined. The most practical and effective method suggested has been given by Fink and Rohrman (271) who found that cobalt can be removed completely by employing rapid circulation of the catholyte, a high pH and a current density of 1 to 1.5 amps./sq.dm. The rapid circulation is the particularly vital factor, which distinguishes this electrolytic purification process from usual low current density purification processes. Two Russian articles of more recent origin by Shcherbakov, Loshkarev, and Lochkarev (577), and Fesin, Andreeva, Moreno, and Schmanzar (608) indicate that they have developed processes for freeing nickel electrolytes of cobalt by the use of sodium hypochlorite and sodium hydroxide, although the exact purification procedure is not available. It is understandable that little has been published on the removal of cobalt from nickel plating solutions primarily because cobalt does not seem to cause any objectionable effects, and in a large number of cases where it is present in the bath, it is there because it was intentionally added. The extreme similarity of cobalt to nickel will undoubtedly make any separation of the two metals difficult, and this factor probably accounts for the lack of information on the separation.

COPPER

The Effects of Copper in Nickel Plating Solutions: It was a well-known fact for many years that copper had an adverse effect on nickel plating solutions, but the first investigation appears to have been carried on in the laboratories of "Brass World," in 1911. (26) At the time of this investigation the voltmeter was the important electrical control instrument, so that the results are not easily interpreted in the light of modern concepts of control. They found, however, that the addition of 1/16 oz./gal. of copper sulfate to a pure nickel plating solution, using 90-92% anodes, caused a darkening of the deposit at 3 volts, and at 2 volts, a less intense darkening in the same length time. The addition of 1/8 oz./gal. of copper sulfate caused almost instantaneous darkening at 2 volts, and immediate darkening at 3 volts. When the copper sulfate addition was increased to $\frac{1}{4}$ oz./gal., the black deposit formed immediately, and a smut formed on the surface of the cathode which could be easily wiped off. When this deposit was buffed, it had all the appearances of a burnt deposit. The addition of 1 oz./gal. of copper sulfate caused immediate deposition of the black smut, and the deposit was black and non-adherent. It was also noted that the anodes turned to a slight copper color, on standing in the bath with no current.

Late scientific investigation on the effect of copper was made by Thompson and Thomas (156) at the Bureau of Standards. Although their investigation was primarily devoted to determining the permissible limits of impurities in nickel salts, their results gave valuable information on the effect of metallic contaminants in various concen-

trations. Zero to 0.25% copper in the nickel sulfate, used to make up the plating bath, showed no ill effect. When the concentration reached the limit of 0.50% the deposit showed a few dark trees near the bottom edge. At 0.10% copper the deposit was treed over with a dark, spongy deposit along the bottom. At 0.50% copper the deposits were similar to those at 0.10%, but more exaggerated. Over 0.50% copper in nickel sulfate produced spongy gray deposits on the bottom of the cathode. Thompson and Thomas also found that the 90 to 92% anodes which were in use at that time might contain 0.10 to 0.25% copper, but that most of this copper fell to the tank bottom as sludge.

Two years after Thompson and Thomas reported their observations on the effect of copper in nickel sulfate upon the deposits produced, Haring (179), also working at the Bureau of Standards, found that copper increased the throwing power of the nickel plating solution. It also produced dark spongy deposits at low current densities, but has little effect on appearance at high current densities.

Setlik (307) has also reported the effect of copper at 3 concentrations in a boric acid buffered double-nickel sulfate bath. At 0.243% copper in the bath the deposit was stained, while at 1.266% copper the deposition of nickel ceased completely. At 2.55% copper the copper precipitated as a powder.

The latest information on the permissible limits of copper as an impurity has been given by Diggin (664). He considered that in dull baths producing ordinary thicknesses of nickel, up to 0.5 oz./gal (0.38 g./l.) of copper can be tolerated. In bright nickel plating baths the upper limit is about 0.001 oz./gal. (0.0075 g./l.). Bright nickel

plating baths have, of course, considerably lowered the permissible limit of copper as an impurity. Eckelmann (247), Johnson (448), and Francis-Carter (506) have pointed out that the two main effects of copper in bright baths are the appearance of dull, dark deposits in low current density regions, and fogging of the deposit in other regions.

The permissible limit of copper in anodes was thought by Thomas and Blum (189), (190) to be about 0.25%. Above this figure, they felt that the deposit would be affected, but below 0.25% the copper would go into the sludge.

While copper in small amounts causes extremely bad deposits of nickel, a great many alloys of copper and nickel have been successfully deposited. Black nickel solutions containing copper have been reported by Watts (54) and others. Copper-nickel alloys have been electrodeposited by Hineline and Cooley (201), Stout, Burch, and Langsdorf (289), Wilmer (311), Kurrein (316), Stout (330), Parlon and Le Baron (426), and Lainer and Shatunovskaya (481). Iron-copper-Nickel alloys have been deposited from sulfate-borocitrate baths by Stout and Faust (328), and by Paweck, Bauer, and Dienbauer (356), who deposited an alloy similar to monel metal. Izgaryshev and Ravikiovich (518) have also deposited a copper-zinc-nickel alloy.

It can be generally said that the effect of copper in low concentrations is to produce a dark, dull deposit particularly in low current density regions. 0.5 Oz./gal copper is the upper limit permissible in most dull nickel solutions, while 0.001 oz./gal. is the limit in most bright nickel solutions.

The Removal of Copper from Nickel Plating Solutions: Since copper is lower than nickel in the electromotive series, it is expected that nickel would precipitate copper from plating solutions. Under proper conditions this principle has been the basis for a number of proposed schemes of purification. One proposed by Thompson and Thomas (156) in 1922 is still used to some extent today. They suggested that the contaminated bath be slightly acidified, and then scrap nickel or scrap iron be allowed to hang in the bath. The efficienty of purification by cementation of the copper would obviously depend on the surface area of metal exposed. Peck and Knittel (318) proposed the use of a powdered metal, largely nickel, while Gindlin (511) proposed the use of finely divided iron. Fink and Rohrman (270) have pointed out that the copper removal was dependent upon pH, temperature, agitation, amount of metal surface exposed, and the concentration of the copper. Although a pH of about 5 and a high temperature are the best conditions. The elimination of copper is presumably never complete.

Basitch (281) proposed a process of purifying nickel plating baths by circulating the polution over nickel hydroxide. The concentration of copper and other metallic ions are reduced to a very low value by this process.

Although some copper can be removed by the Liscomb ferric hydroxide precipitation method (330), the removal is far from complete. The most satisfactory method yet proposed for commercial purification of nickel plating solutions is by selective electrolysis at a low current density. Although this principal was known for a long time, it is not known definitely when it was first applied to nickel plating solutiond. Fink and Rohman (270) in 1930 found that high temper-

ature, rapid solution agitation, and low current density (0.10 amp./sq.ft.) would remove more than 90% of the copper. In a later article (271), Fink and Rohrman found that the last traces of copper could be removed by rapidly rotating the cathode in the solution while electrolyzing at a low current density. Since these two articles were written, many authors have suggested this means for removing copper-Raub & Bihlmaier (392), Weisberg (468), (541), (582), Mattacotti (484), Javeli (519), Smith (538), Stocker (540), Diggin (664), and Waite (666). According to Weisberg (582) copper and lead are most efficiently removed at 1 or 2 amps/sq.ft..

A process for refining nickel in the presence of copper and iron was proposed by Guess (115) in 1919. In this process the electrolysis took place in a suspension of calcium carbonate, with a little glue also added. The copper was evidently not removed from the solution, but its deposition prevented. Separation of trace amounts of copper from solutions, particularly water solutions, has been the subject of several articles which may prove helpful in further work along this line. In 1918, Saul and Crawford (110) found that Quinsol separated copper from solutions. The mercuric ion is the only ion which interferes wit this separation. Adams (266) found that flauconite removed completely small amounts (100p.p.m) of salts of lead, zinc, copper, and tin. Brockman (387) has shown that copper ions could be removed in phosphate buffered solutions at a pH greater than 5.3 by sodium aluminate, providing the aliminate/copper ratio is greater than 6.5. Holl (379) has also been successful in removing lead and copper from water by passing it through a magnesium oxide filter.

FLUORIDE

The Effects of Fluorine in Nickel Plating Solutions: In 1908, Kern and Fabian (18) published results which they had found in plating nickel from fluosilicate solutions. This article and one by Kern (22) the following year appears to be the first use of fluorine compounds in nickel plating solutions. The fluosilicate bath was found to produce bright, non-crystalline, tough, flexible, and adherent deposits. Silica was found to settle out during deposition, but did not interfere with electrolysis. Excellent results were also claimed for the fluoborate solution, and in both cases, it was found that the impurities in the anodes remained on the anodes. Hollard (49) and Sperry (44) also proposed the addition of hydrofluoric acid with boric acid, as a means of obtaining thick deposits of nickel, and preventing the liberation of hydrogen at the cathode. In 1920 hydrofluoric acid additions received new attention when Lovering wrote an article (123) on his "magic fluid." The claims which Lovering made for the addition of hydrofluoric acid and fluoborate additions were, that excellent anode corrosion was produced, anode sludge formation was prevented, and cathode efficiency was increased. Blum (128) then undertook scientific,investigation of fluoride and fluoborate additions and reported that when nickel fluoride or sodium fluoride is added to nickel baths containing boric acid, fluoborates more complex than those corresponding to the simple formula of fluoboric acid are probably produced. He found, furthermore, that the nickel deposits produced from solutions containing fluorides had finer structure and greater tensile strength than those from corresponding chloride solutions. Also, when anodes of 97% nickel were used, the deposits

produced from the fluoride solution contained slightly more iron and copper than those from the chloride solution. The anode corrosion in the fluoride solutions wa also found to be good, and less sludge was formed in the fluoride solutions. Blum together with Slattery (129) also found that higher current density and greater tensile strength were possible from solutions containing fluorides. Watts (144) also investigated fluoride additions the same year as Blum and reported that a nickel fluoborate solution, either alone, or with glycerine also ad ed, would give nice deposits. A mixture of fluoborate and glycerin produced remarkably smooth deposits, which were also bright, but brittle. Watts also pointed out that both hydrofluoric acid and boric acid are weak acids from the dissociation aspect, and therefore can be added in large quantity without detrimental effects, and will also keep the solution acid for some time. He also found that the anode corrosion was increased, and the resistance of the solution and efficiency of deposition were lowered. Watts also felt that the beneficial effects of hydrofluoric acid additions were due to the formation of a fluoborate. This latter fact is an important one since the hydrofluoric acid was rarely added to a solution not containing boric acid, so that the effects reported are probably due jointly to boron and fluorine. Woodmansee (146) confirmed Watts observations and also found that more voltage was required in the fluoride solution over the chloride solution, and the resulting deposits were harder to buff.

In 1924, Hammond (178) made a study of the effects of the addition of conducting salts, and one of his observations was that the addition of sodium fluoride reduced the resistivity of the solution. Haring

(179) the same year found that replacing ammonium chloride in a nickel plating solution with sodium fluoride reduced throwing power. Haring's observation together with Hammond's, that ammonium chloride additions had the best effect on the character of the resulting nickel plate, helped bring about a change of opinion on the effects of fluoride additions. Pleadwell (140) had earlier found that the tarnishing of nickel-plated ware on heating was not due to the copper undercoat, as had been supposed, but due to the codeposition of iron with nickel. He found that the hydrofluoric acid additions allowed more iron to be deposited. Thomas and Blum (189) then found that fluorides did not materially increase anode corrosion, and Dorrance and Gardiner (241) found that the fluoride ion does not materially reduce anodic polarization. Hogaboom (255) also found that sodium fluoride additions cause loose particles to form at the anodes, with possible resultant rough cathode deposits. Macnaughton and Hothersall (232) in investigating the effect of various additionson the hardness of nickel deposits found that sodium fluoride did increase hardness somewhat, but that potassium chloride had the same effect. They also pointed--"A serious drawback to the use of solutions containing fluorides is the low anode and cathode efficiency."

Fluoride additions enjoyed brief popularity, but occasionally some good effects were reported. Ollard (219) reported that fluoride additions produced marked buffering effects in both the absence and presence of boric acid. He also found that the fluorides helped keep iron in solution, which resulted in even, fine-grained deposits, and decreased pitting. In contradiction to other investigators, Ollard found that fluorides produced a softer deposit. Ollard noted that

organisms such as Penicillium glucinium have actually been found growing in nickel plating solutions, and that the presence of fluorides seemed to prevent their growth.

The use of fluorides came into practice more recently when Loose (630) found that magnesium alloys could be successfully nickel plated in a nickel fluoborate solution. The bath contained a slight excess of boric acid and 3 to 7 g./l. of fluorine over that required to form nickel fluoborate. Except for this special case, fluoride additions have been generally ignored, although nickel fluoborate solutions along with other metal fluoborates have been receiving some attention. The high cost of the initial bath seems to prohibit their general use, although they are presumably little more expensive to keep constant than the regular nickel sulfate baths.

The Removal of Fluorine from Nickel Plating Solutions: There are apparently no published methods for specifically removing fluorides from a nickel plating solution. It is quite probable that their removal has not been necessary since they seem to produce no noticible effects.

GOLD

The Effects of Gold in Nickel Plating Solutions: The probability of gold occurring as an impurity in nickel plating solutions is very small. The few published articles on the subject deal mainly with gold-nickel alloys. Raub and Bihlmaier (458) in 1937 found that when nickel was added to a gold bath so that the bath contains 1 gram of gold and 1-5 grams of nickel per liter, a fine white gold plate of about 15% nickel composition was produced. Young (591) has also written an article in which gold-nickel alloys are discussed. Probably the closest approach to determing the effect of gold in nickel plating solutions was made by Puri and Alvi (571) who found that small additions of a gold sol had beneficial effects on hardness and luster of the plate, and the pitting tendency was reduced. Even in this case it must be assumed that the results observed were due to the colloidal properties of the gold sol, rather than due gold itself, although the gold may have had a contributing influence.

The Removal of Gold from a Nickel Plating Solution: Gold could be removed from a nickel plating solution by electrolysis at a low current density. Rapid solution circulation or a rotating cathode would also facilitate its removal.

HYDROGEN

The Effects of Hydrogen in Nickel Plating Solutions: It is difficult to consider hydrogen as an impurity in nickel plating solutions because it is probably always present unless some special means is taken to prevent its formation. The phenomena of hydrogen embrittlement of base metals and deposits is well known, but such a discussion is beyond the scope of this paper. Hydrogen has been attributed to be the cause of several effects, which should be considered. Stockmeir (19) considered hydrogen as a cause of peeling of nickel deposits, and Forstner (226) has pointed out that heavy hydrogen and heavy water which are known to form in some plating solutions might be a cause of some plating difficulties. Although Forstner has no experimental results to present, his theory is unique. One of the most common effects attributed to hydrogen is pitting. It is now generally considered that pitting is of several different types depending upon the different causes. Hydrogen of itself is not considered to be a cause for pitting, but when hydrogen bubbles collect on grease or other foreign matter on the surface of the plate, they may be a secondary or contributing cause of pitting.

Kohlschutter (181) found that eliminating the film of hydrogen at the cathode produced a coarsely crystalline deposit. He found also that abnormal polarization was largely eliminated and that there was little or no contraction.

The Removal of Hydrogen from Nickel Plating Solutions: In the case of hydrogen in nickel plating solutions, the "impurity" is not removed, but converted to a harmless form. This process probably involves the

oxidation of the hydrogen, the instant it forms at the cathode surface, by an oxidizing agent. Hydrogen peroxide, sodium perborate, and boric acid are among the compounds used for this purpose. In modern, organic-type bright nickel solutions these oxidizing agents are not compatible with the brighteners, so that wetting agents are generally used instead. Wetting agents lower the surface tension of the solution so that it is difficult for hydrogen to form and collect at the cathode. Oxidizing agents and wetting agents will be further discussed as impurities in a later section.

IRON

The Effects of Iron in Nickel Plating Solutions: The effects of iron as an impurity in nickel plating solutions are perhaps the most widely known and studied of all the metallic impurities. The reason for this is quite apparent since iron and nickel, being chemically similar, are practically always found together. In the early part of this century when 90-95% nickel anodes were used, the residual 8-10% of the anodes was largely iron. This gave rise to sludge formation and the codeposition of appreciable quantities of iron with the nickel. At this time there was quite a diversity of opinion as to the actual effects of iron, and there was some argument as to whether it was actually a beneficial or harmful contaminant.

One of the more commonly observed effects which was reported was that iron in the nickel plating solution caused the resultant deposit to be smoother and whiter in color. This view was confirmed in the "Brass World" laboratories (30), and shared by Proctor (150), Thompson (172), Barrows (214), and Clindinin (295). Thompson also reported that the ratio of iron to nickel in the deposit was higher than that in the solution. The codeposition of iron (2 to 7%) with nickel at a constant pH was found to give a more finely crystalline deposit, but the alloy thus formed was also harder, more brittle and under more internal stress.

There appears to be some disagreement on the color of an iron-contaminated nickel deposit. Though many reported that the deposit was lighter, Setlik (307) reported that, while 0.10 to 0.40% iron in a

nickel bath showed no effect, 0.604% iron gave a dark deposit, and 2.02% iron left unplated areas particularly in low current density regions, and the deposits were dark and irregular. In other cases where dark deposits have been reported as due to iron contamination, the viewpoint has been that iron-containing nickel deposits tarnished more easily (20). Mathers, Stuart, and Sturdevant (81) in trying to overcome sludge formation in nickel baths, found that the use of ammonium citrate gave dark deposits containing 6.23% iron from anodes containing 6.49% iron. The deposits from solution containing m. citrate additions gave deposits containing 3.31% iron. The citrate thus increased the co-deposition of iron with nickel, and the deposits which resulted were of a bluish, or a dark color. The use of citrates or citric acid for preventing sludge formation was also discussed by Barrows (46), Later (99) and Mathers (257). Mathers (257) and Pleadwell (140) also mentioned the use of hydrofluoric acid for the same purpose. In the light of the findings of Mathers, Stuart, and Sturdevant (81), however, it hardly seems that it is advisable to keep the iron in solution. One could argue, however, that the many ill effects due to sludge and suspended iron hydroxide are much worse. Since high-purity anodes are practically exclusively used today, either argument appears weak.

It has been widely reported that suspended particles give rise to rough, hard, cracked deposits. One of the biggest sources of suspended particles in a nickel bath is iron hydroxide. This precipitate is gelatinous in form and can easily remain suspended in solution, or can migrate through the bath by thermal and electric currents. When these particles are co-deposited, they can cause rough deposits, and also ones which are hard and highly stressed or cracked. Such

observations have been reported by Voss (191), Evlanikov and Neiman (506), Mattacotti (527), and by Morkhov, and Atchi (566) who also found that the iron to nickel ration was four times greater in the deposit than it was in the solution. They also considered the permissible limit of iron to be 0.2 g./l. Eckelmann (347), Johnson (448), and Shepard (220) reported that, when the ferric hydroxide particles settled on the cathode, pits could be formed. Shepard (220) also found that when the iron was in the ferrous state, there was a tendency for smoother deposits to form, and also for pitting to diminish. Heil (65), Kohlschutter and Vuilleumier (106), Schlotter (142), Vuilleumier (143), (158), (223), (240), Thompson (172), Raub (391), and Martin (657), all recognized that iron,contamination gives rise to stressed deposits, which also may cause peeling if the stress becomes great enough. Vuillenmier (158), showed that even a small quantity of iron would increase the contractive effect of nickel deposits by 50 percent. Raub (391) also found that keeping the pH below 4 prevented peeling when the iron content is high. Martin (657) found however, that in low pH Watts type solutions both ferrous and ferric iron increased stress, while at a pH of 5.5, ferrous iron reduced stress.

Often ferric hydroxide acts like colloidal particles, and as such may give rise to brighter, smoother plates, but also ones which are hard, brittle, and even "burnt". Romanoff (358) has written that ferric hydroxide is the most common of the "electro-positive" colloids, and may cause pitting. Puri and Bhatia (533), and Puri and Juneja (570) have added ferric hydroxide sols to nickel plating solu-

tions and reported that luster was increased, as w s hardness.

There appears to be some disagreement in the two cases where the effect of iron on current efficiency was investigated. Bennett, Kenny, and Durliss (59) felt that the iron content of anodes did not materially affect the efficiency, but Mathers and Sturdevant (62) in an article two years later contended that the iron present in 90-92% anodes was sufficient to lower efficiency. In either c se the effect is not very large, and in the use of high-purity anodes, as is the practice today, it is doubtful that enough iron can accumulate in the solution to materially lower current efficiency.

Since the advent of bright nickel baths, a new effect has been observed which has been attributed to iron contamination. This effect is that iron causes dulling, or blushing of the deposit, and has been reported by Weisberg (496), Francis-Carter (508), and Meyer (607). The dulling of a bright deposit is a rather general effect not only from iron contamination but also from copper, lead, and zinc contamination, as well as from numerous organic materials.

Roberts (429) observed that good deposits of nickel could not be obtained on cadmium or zinc-plated steel in the presence of ferric iron, even when a pre-strike was used. He also found that large additions of magnesium sulfate did not remedy the situation.

It is obvious that the chemical similarity between iron and nickel, and the ease with which they are deposited, would lead to the possible electrodeposition of a great variety of iron-nickel alloys. The bibliography on the deposition of nickel-iron alloys is quite sizeable in itself. A list of this sort would list articles by Kuster (1),

Bruni and Amadori (47), Sucky and Haas (52), Kremann and Haas (60), Benvenuti (75), Kremann and Breymesser (98), Glasstone (162), Glasstone and Symes (227), (242), Wilmer (311), Murrein (318), Stout (329), Stout and Faust (338), Marshak, Stepanenov, and Bolyakova (355), Paweck, Bauer, and Lienbauer (356), Marshak, Stepanov, and Levius (387), Raub and Walter (393), (394), Skalozubov and Goncharova (578), and DuRose and Pine (656).

In general the effects due to iron can be summarized as follows: The deposit will tend to be whiter, but more subject to tarnishing and corrosion, and will be harder, more brittle, and under more stress. The permissible limits for iron in modern nickel baths is thought by Diggin (664) to be 0.03 to 0.22 oz./gal..

The Removal of Iron from Nickel Plating Solutions: Several factors have been developed from time to time which have materially added the removal of iron from nickel plating solutions. One of the first of these factors was one noted by Calhane and Gammage (9) in 1907. These men found that agitation of a nickel solution contaminated with iron caused the deposition of iron to be 3 to 5 times greater than in a stagnant solution under the same current conditions. Guess (115) described a method of electrolytically refining nickel in the presence of iron and copper by using a suspension of calcium carbonate to which a little glue had been added. It is only recently that much has been done on the electrolytic method of removing iron from a nickel bath. Weisberg (468), (541), and Diggin (664) have found that electrolysis at a cathode current density of 5 amps./sq.ft. would successfully remove iron and zinc.

The most practical and successful method of removing iron has been by the precipitation method. It can not be definitely stated when this method originated. Since the early nickel baths were operated at a pH above that at which ferric hydroxide precipitates, the removal of iron by this method was actually carries on automatically. A large amount of the "sludge" which received so much attention in the early days was ferric hydroxide, and this precipitate settled to the bottom of the tank and was removed periodically. The first precipitation methods employed the use of nickel carbonate (111), or nickel carbonate and barium peroxide (157), or magnesium carbonate and ammonium persulfate (172). Then Fink and Rohrman (271) in preparing pure electrolytic nickel found that, after removing most of the iron by electrolysis, the remainder could be removed by adding ammonium hydroxide until the iron precipitated and then aerating the solution for twenty-four hours, followed by filtration. Although Liscomb's (330) method was primarily used for removing other metallic impurities by carrying them along with the ferric hydroxide precipitate, the method ovbiously also removed iron. Gutman and Mayantz (349) modified the precipitation method a little by first oxidizing any ferrous iron to ferric iron by using manganese dioxide and then treating the solution with magnesium or calcium carbonates to precipitate both iron and aluminum Zakharova, Kisharova, Baumn, and Galendeev (363) found they could remove iron from nickel sulfate solutions by adding sodium carbonate and then aerating the solution to oxidize any ferrous iron to the ferric state. Costello (370) has recommended nickel carbonate treatment of the solution to raise the pH

to a value to precipitate the iron. This method of raising the pH of the solution has gained considerable favor since no extraneous cations are added to the solution, which prevents the ill effects sometimes attributed to the alkali metal ions. However, as recently as 1939, Gindlin (511) has suggested removal of iron by treating the solution with hydrogen peroxide and sodium hydroxide, and filtering to remove the precipitate. Jeveli (519) and Smith (538) have also recommended the precipitation method as has Stocker who suggested raising the pH of the solution to 6.0 with lime and nickel carbonate, then adding hydrogen peroxide, and finally filtering. The most recent recommendation has been made by Diggin (664) who has given the following instructions: "Raise pH of the bath to above 4.7 (electrometric), add one pint, per 100 gallons of solution, of 100 volume hydrogen peroxide to the hot solution and filter."

These two general methods are the most common methods which have been, and are being practiced for removing iron from nickel plating solutions. However, several suggestions have appeared in the literature, which are interesting. Berthelot and Gaudechon (25), for instance, found that iron was precipitated as ferric hydroxide when subjected to the action of ultra-violet rays in the presence of nickel or cobalt. A patent by Grönningsaeter (317) indicates that iron can be removed by the action of powdered activated nickel. Wille and Völkel (438) have a German patent for removing iron from solutions of nickel and cobalt by the use of alkali perphosphate. Samuel (575) has a British patent for removing iron by adding cellulose pulp, nickel or cobalt peroxide, higher oxide or hydroxide, to the bath, and filtering. Shepard and Knierim (658) have recently obtained a

patent for removing iron which stipulates adjusting the pH of the solution to 3-4, heating to 80-180°F, oxidizing with hydrogen peroxide, and then maintaining the pH by the addition of calcium hydroxide. The calcium hydroxide and hydrogen peroxide are added to the solution as a slurry.

LEAD

The Effects of Lead in Nickel Plating Solutions: In 1924 Shepard (186) (209) recommended that a brightener for nickel plating solutions could be made by adding acetic acid to a 20% lead acetate solution until it was clear, and adding 3 oz./gal. of this solution together with ½ oz./gal. of glycerine. Excellent results were claimed for this brightener. When the plate began to dull after some time of operation, 1oz./100 gal. of the 20% lead acetate solution was added to brighten the deposit. Raub and Wittum (459), and Wittum (469), (508), foundthat lead caused fine-grained lustrous deposits, which were bright and brittle with a tendency to peel. They also noted that when lead sulfate was precipitated, the deposits were dull, and rough spots were likely to occur. Cosgrove (408)also reported that lead contamination caused non-adherence of the deposit, and also noted that this did not appear until chrome plating was practiced. Ballay (344), Mattacotti (527), Meyer (529),(607), and Stocker (540) found that lead gave rise to dark, streaked deposits, and the effects were most noticeable in low current density regions. Martin (657) found that in a low pH Watt's type bath, lead in low concentrations had little effect on the stress of nickel deposits, but at a pH of 5.5 lead reduced stress. In bright nickel solutions Eckelmann (347), Johnson (448), and Francis-Carter (508) have reported that lead contamination caused dark streaks to appear. In nickel-cobalt bright nickel solutions Weisberg (496) has found that under average conditions 5 mg./l. of lead revealed dark portions at the band of bent cathodes. Diggin (664) has also reported that while lead was only

slightly soluble in nickel solutions, the presence of citrates, acetates, and formates held lead in solution, and he considered the top permissible limit of lead to be about 0.002 oz./gal..

Since the permissible limit of lead is very low, lead is one of the worst contaminants of nickel solutions. Lead plumbing and lead tank linings have been a frequent source of lead contamination. The effects due to lead can be summarized as causing brittle deposits with a tendency to peel under chromium deposits. The deposits also tend to be dark in low current density regions, and dark streaks may appear in normal current density regions. Under certain conditions, very smooth and bright regions may appear in the deposit.

The Removal of Lead from Nickel Plating Solutions: The removal of lead from nickel baths does not produce any difficulties. Wittum (469) proposed that lead could be removed by strongly acidifying the solution with sulfuric acid, and heating it in order to form insoluble lead sulfate. The solution was then cooled, filtered, and the pH restored. The trouble with this method would be the difficulty in restoring the pH, which would require quite some quantity of nickel carbonate. The most practical method has been proposed by Stocker (540), Mattacotti (484), Weisberg (541), and Diggin (664), who proposed low current density electrolysis at 2 to 3 amps./sq.ft., together with a pH of about 2, and a fairly high temperature.

MAGNESIUM

The Effects of Magnesium in Nickel Plating Solutions: A great many effects have been attributed to the presence of magnesium in nickel plating solutions. During the period of 1910 to 1920 magnesium sulfate additions were quite popular, and as a result, its effects were well investigated. One of the first effects noted was that the plate which resulted from a solution containing magnesium sulfate had a yellowish tinge to it. This was reported simultaneously by Sizelove (88) and Taylor (89). Proctor (60) had earlier reported the use of magnesium sulfate as a grain-refining or brightening agent. More recently Makar'eva (482) reported that deposits resulting from the addition of magnesium sulfate were more lustrous, harder, and more uniform in texture. Evlampikov and Neiman (506) in an effort to produce nickel plate free from pinholes found that magnesium sulfate in quantities from 1 to 100 g./l. produced matte, white deposits. Another report by Walters (102) pointed out that a small amount of magnesium sulfate added to a nickel bath produced a more ductile plate, but if it was added in excess, the deposit might be darkened. Richards and Mathiges (208) added magnesium sulfate in amounts from 4 oz./gal. to 16 oz./ gal. to nickel solutions containing 1 lb./gal. of nickel sulfate and 2 oz./gal. of boric acid. They noted that the deposits were good and white at all concentrations, and in a range of current densities from 6 to 15 amp./sq.ft. there was slight gassing at aconcentration of 16 oz./gal, but no pitting was noted in any case. Diggin (664), (596) has reported that the presence of magnesium sulfate in the solution made the resulting deposit much

softer and easier to buff. In a nickel-cobalt bright nickel he found the Vicker's hardness to be reduced from 508 to 398 by the addition of 2 oz./gal. of magnesium sulfate. Diggin (597) also studied the effect of substituting magnesium chloride for nickel chloride in standard baths and reported favorable results up to a concentration of 3 oz./gal. Above this concentration the deposit developed hair-line cracks. Two separate investigations have been made to determine if any magnesium is co-deposited with nickel, if the solution contains magnesium sulfate. In one of the first articles on the use of this salt, Wäser and Schulz (45) found that 1.5% magnesium was co-deposited with the nickel. In 1938, however, Bulalsh (475) investigated this problem and reported no magnesium in the plate. In the light of what has been learned since 1912, the observation of Bulalsh, is undoubtedly correct.

Many effects have also been reported on the effects of magnesium sulfate additions on the operation of nickel plating baths. Hammond (178), Underwood (221), and Diggin (664) have reported that it increased bath conductivity. Magnesium sulfate also increases the throwing power of nickel plating solutions, according to Haring (179), and Diggin (596). Mathers, Stuart, and Sturdevant (81) reported the use of magnesium chloride to prevent anode sludge formation, and a similar report was made by Robinson (87), using magnesium sulfate. Robinson also noted that the anode surfaces remained quite clean, and any trouble due to pitting was removed.

Magnesium sulfate additions have received much favor in plating non-ferrous materials. Proctor (51) advocated the use of magnesium sulfate additions to nickel plating solutions when die-castings were to be plated.

Herrick (148), Thompson (312), and Gordon (274) have since confirmed the value of this addition. Proctor (118) also pioneered in the use of magnesium sulfate in nickel solutions used to plate aluminum. Holland (165) found that magnesium chloride could also be used, but Foss (252), Werner (291), Krause (328), and Ballay (342) have preferred the use of magnesium sulfate. Underwood (263) has also recommended the use of a solution containing magnesium sulfate, ammonium chloride, and cadmium chloride for bright plating small lead parts.

The Removal of Magnesium from Nickel Plating Solutions: No information has been found on the removal of magnesium from nickel plating solutions. Magnesium is not a very probable accidental contaminant, and its presence is generally attributed to intentional additions for special purpose plating. The effects of magnesium are, as a rule, not objectionable; however, if a method of removal is desirable, such investigation will be undertaken in connection with this research.

MANGANESE

The Effects of Manganese in Nickel Plating Solutions: Very little has been published on the effect of manganese in nickel plating solutions. Barrows (214) and Meyer (454) have pointed out, however, that manganese is sometimes added to deoxidize molten nickel before it is cast into anodes. They pointed out that if manganese gets into the solution rough deposits may result. Potassium permanganate has been frequently suggested as an oxidizing agent to overcome organic contamination of nickel plating baths. Shepard (188), (220) and Romanoff (267) have overcome the ill effects of organic contamination in this manner. Shepard also found that the potassium permanganate additions counteracted the hardening effect of cadmium as a brightener. The argument for the use of potassium permanganate over other oxidizing agents in overcoming organic contamination was that the permanganate acted as its own indicator, denoting when enough had been added. If an excess of permanganate is added to the solution a precipitate of manganese dioxide is formed, which will eventually dissolve in the bath. Thomas and Blum (210) found that permanganate additions did not markedly decrease porosity. Since 1930 it does not appear that the use of potassium permanganate as an oxidizing agent in nickel solutions has enjoyed much popularity. Since the reduction product of hydrogen peroxide is simply water, it would obviously be preferable. Campbell (174) showed that it was possible to form alloys of manganese and nickel by electrodeposition. In certain concentrations, it would appear that manganese is not harmful in nickel solutions, but a real source of information on the effects of manganese in various concentrations is not available, nor has work been done on its removal from nickel plating solutions.

MERCURY

The Effects of Mercury on Nickel Plating Solutions: There are two published accounts of the presence of mercury in nickel plating solution. The first account was made by Watts (192) in 1924, who tried the addition of mercuric chloride in an effort to prevent the formation of hydrogen at the cathode. The result of the addition was that the depolarizing action was so strong that no nickel would deposit. In the other account Solanki and Singh (613) added trace amounts of mercuric chloride to a nickel plating solution, and observed that the deposits which resulted were loose and powdery. From the results of these two cases, it is obvious that mercury contamination of a nickel plating solution would either totally inhibit deposition or produce entirely unsuitable deposits.

MOLYBDENUM

The Effects of Molybdenum in Nickel Plating Solutions: In 1920, Barrows (121) added ammonium molybdate to a nickel sulfate solution containing magnesium sulfate and boric acid, and reported that a black deposit was obtained. Since black deposits form from solutions containing molybdates, it is rather certain that molybdenum in nickel plating solutions would lead to black or dark colored deposits. In the only other account on the effect of molybdenum in nickel solutions, Furi and Seth (612) found that a molybdenum sol added to a nickel sol tion imparted luster to the deposit. Here again is a case where the colloidal properties of the sol may be the contributing factor to the observed results.

NITRATES

The Effects of Nitrates in Nickel Plating Solutions: The effects of nitrates in nickel plating solutions have been known for some time. In 1913 Sumec (53) found that nitrates or nitric acid would cause dark deposits. Mathers and Sturdevant (82) also found that nitrates, which may occur as an impurity in nickel salts, depressed the current very strongly. Madsen (136), and Watts (193) found that nitrates were such strong depolarizers that they could easily cause deposition of nickel to cease completely. Nichol and Watts (204) have reported that the amount of nitrates necessary to cause no deposit to form was in the neighborhood of 40 g./l. According to Eldridge (212), however, M. Bailey of France recommended the use of small amounts of nickel nitrate as an oxidizing agent. An excess of the nitrate was reported to cause fragile and burnt deposits, as well as a rapid drop in the current efficiency. Rothersall and Hammond (371), (380) then undertook a study of the effect of nickel nitrate on nickel deposition and found that it increased hardness and stress, and brightened the deposit. Since the deposit was stressed, there was also a tendency for the deposit to become cracked and exfoliated. As electrolyses continued the nitrate was reduced to ammonia which in turn prevented the precipitation of nickelous hydroxide. With increasing concentration of nitrate the cathode potential was gradually noted to fall, and the amount of hydrogen released at the cathode decreased. At a concentration of 2.6 g./l. the formation of hydrogen ceased entirely. As nitrate was added to the solution an initial increase in pitting was also noticed, but then it gradually decreased with further nitrate

additions, and finally ceased when hydrogen formation also ceased.

Nitrates have not found very extensive use as oxidizing agents, primarily since hydrogen peroxide produces better results, and is easier to control. Eckardt (346), (372) has recommended baths for depositing non-porous and hydrogen-free deposits, which contained sodium nitrate, but the composition of the baths is so complex that control would be very difficult. Fadeeva (551) has more recently investigated the use of nickel nitrate in solutions being operated that the addition did not decrease porosity. This viewpoint has also been shared by Diggin (596), (664) who found that nitrates not only increased pitting, but lowered cathode efficiency, and decreased throwing power. In cobalt nickel baths, the nitrates were also found to restrain the deposition of nickel.

The Removal of Nitrates from Nickel Plating Solutions: The removal of nitrates from nickel plating baths becomes evident from the observation of Hothersall and Hammond (351), that nitrates are reduced to ammonia during the operation of the bath. The best operating conditions have been suggested by Diggin (664) who proposed that the bath be made acid with 2 cc./gal. of sulfuric acid and then operated using dummy copper cathodes.

HYDROGEN PEROXIDE

The Effects of Hydrogen Peroxide in Nickel Plating Solutions: Hydrogen peroxide is added to nickel plating solutions for two purposes. The primary purpose is as an ingredient of nickel baths for preventing the deposition of hydrogen at the cathode. Stiger (125) was the first to study this addition when he added hydrogen peroxide as a depolarizing agent to the solution and noted that the normal stress on the deposit was reduced. Madsen (182) was the first to recommend this addition in regular practice. He recommended a daily addition of 4c.c./l. of 3% peroxide, or a concentration of 0.12g./l. of nickel solution. Haring (100) found that peroxide in this concentration reduced the throwing power of the solutions, and Blum and Thomas (210) found that peroxide also promoted anode passivity. In 1934, Mothersall and Hammond (251), (250), undertook an investigation of the effects of hydrogen peroxide on nickel deposition, and their report has been most valuable. Additions of peroxide were found to increase hardness, stress, and the bright appearance of the deposit. The deposits thus tended to become cracked and exfoliated, and this was attributed to the precipitation of larger amounts of basic colloidal matter with the rising rate of hydrogen ion discharge. With increasing additions of peroxide, the amount of hydrogen released at the cathode became gradually reduced to zero, but on further additions the gas discharge recommenced. They attributed this observation to the reduction of the nickel ion concentration at the cathode face as a result of the precipitation of a considerable quantity of basic matter. The cathode efficiency was also observed to be linearly decreased with increasing additions of peroxide until a minimum value

was reached which corresponded to zero hydrogen discharge at the cathode. As the additions were increased beyond this critical concentration, the cathode potential showed a marked increase. These authors also felt that any effect which hydrogen peroxide had on reducing pitting was due only to the inhibition of hydrogen discharge. If the solution is impure, however, the action of peroxide in oxidizing and precipitating iron, with coincident adsorption of other impurities by the precipitate, must be taken into account.

The second use of hydrogen peroxide in nickel plating solutions is for removing contamination by iron and organic compounds. A discussion of the effects of hydrogen peroxide in this manner is found under the sections on removal of these impurities.

The Removal of Hydrogen Peroxide from Nickel Plating Solutions: Hydrogen peroxide is very easily removed from nickel plating solutions. Raising the temperature, or the pH of the solution, or a combination of these, will rapidly decompose hydrogen peroxide. Green (51) showed that a a pH of 5.63, hydrogen peroxide was about 97% decomposed in 3 hours. At a pH of 3.7 and a temperature of 65°C. the hydrogen peroxide was 92% decomposed in an hour and a half. A combination of these conditions would probably greatly accelerate the decomposition. The ease of removal of hydrogen peroxide as well as its chemical constitution accounts for its greater application as an oxidizing agent.

PHOSPHORUS

The Effects of Phosphorus in Nickel Plating Solutions: The effects due to phosphorus and its compounds are practically unknown. Blum and Kasper (266), and Cotton (49) have investigated the effect of phosphoric acid as a buffer agent in nickel plating solutions and found it ineffective. Nickel has been deposited from a pyrophosphate solution by Koyangi (337), but this is the solitary case of deposition of nickel from any phosphate solution. Phosphate contamination is possible from cleaning solutions, but if any ill effects result, they are as yet unknown, or unreported.

SELENIUM

The Effects of Selenium in Nickel Plating Solutions: The effects due to selenium are very little known. It is not a probable contaminant, although according to Young (592), selenium may be added in small traces to certain proprietary bright nickel solutions. Puri and Juneja (570) have also found that sols of selenium added to nickel solutions improved the luster and hardness of the deposit. With the exception of several patents, only these two cases have been published on this impurity, and the effect of other than trace amounts of selenium have not been revealed.

The Removal of Selenium from Nickel Plating Solutions: According to Stocker (617) selenium can be removed from nickel plating solutions in two ways. The first method is by raising the pH of the solution to 6.2-6.4, (electrometric) adding nickel hydroxide or nickel carbonate, and an oxidizing agent such as hydrogen peroxide, or potassium permanganate. Selenium will thus be precipitated along with iron, zinc, aluminum, trivalent chromium, and cadmium. Stocker also found that selenium could be removed from nickel plating baths by cementation on iron or nickel.

SILICON

The Effects of Silicon in Nickel Plating Solutions: Kern and Fabian (18) and Kern (22) have contributed the sole references on the effect of silicon in nickel plating solutions. In this case as in the case of some other impurities, it is not the element which is involved but fluosilicate. These authors have reported successful deposits of nickel from a nickel fluosilicate solution. What the effects of silicon or of silicates are, have not been reported. It would appear from this lack of information that silicates have no real detrimental effect in nickel plating solutions, at least in small amounts. This is evident since there is certainly the possibility of silicate contamination of nickel plating solutions from cleaning solutions.

SILVER

The Effects of Silver in Nickel Plating Solutions: Silver is not a very probably contaminant of nickel solutions, and if it were to occur, it would probably not remain in solution in other than very small concentrations. There may be some possibility that silver might be slightly soluble in certain nickel plating solutions, but in those containing chloride, it would probably be largely precipitated as silver chloride. Nickel-silver alloys have been electrodeposited by Behnke (403), and Mathers and Johnson (481) but in neither case was the solution of comparable composition to any nickel plating solution. Puri and Bhatia (533), and Puri and Alvi (571) have added silver sols to nickel plating solutions and found that a beneficial result was produced on hardness and luster. It must be remembered that the silver was present as the element, and not in solution, and that the effects observed may be due solely to the colloidal properties of the sol.

SULFUR

The Effects of Sulfur in Nickel Plating Solutions: Sulfur has been reported as a contaminant in nickel plating solutions in a variety of different forms. Since nickel deposition is done in sulfate solutions, sulfates can not be considered as impurities. In 1908 Turrentine (20) proposed that persulfates could be formed in sulfate solutions by the anodic effect of nickel. A few years later Couteulx (26) found that bright nickel deposits could be obtained from a double-nickel sulfate solution to which ammonium persulfate had been added as a brightener. Sperry (44) also reported a solution of nickel sulfate and ammonium chloride to which had been added sodium persulfate. Another solution Sperry reported contained, besides nickel sulfate, magnesium sulfate, boric acid, and sodium persulfate. Since no ill effects due to persulfates have been reported, and several different baths have been found to yield successful deposits in the presence of persulfates, it is reasonable to believe that they have no ill effect, other than the effects generally attributed to all oxidizing agent, i.e. hard, brittle, stressed deposits when present in excess.

Thiocyanates have been common ingredients in black nickel solutions. Solutions have been suggested by Watts (54), Dellars (76), Blum (105), Proctor (109), Hogaboom, Slattery, and Han (117), (135), Kenna (171), Maxwell (181), Barrows (221), Moline (648), and others, which contained sodium or potassium thiocyanate. The thiocyanates have an important part in producing the black deposit, and Hogaboom, Slattery, and Han (117), (135) have reported as much as 10 to 14% sulfur in black nickel deposits from solutions containing thiocyanates.

Some recent work has been done on sulfamate baths of nickel, with varying claims of success. Cambi and Piontelli (502), and Piontelli and Guilotto (532) claim nickel can be both electrorefined and electrodeposited from nickel sulfamate solutions. Coquill (503) also deposited nickel from sulfamate solutions, but reported that the deposit was not bright.

Sulfides have long been known to be brightening agents in many types of baths. Lenfuelss (627) pointed out that sodium sulfide could be used as a brightener in nickel plating solutions, but the bath is unsatisfactory due to the evolution of hydrogen sulfide and the precipitation of nickel sulfide. Aryl sulfonamides, and aryl sulfonimides as well as polysulfonic acids have been listed by Zimmer, Soderberg, and Baker (609) as being brightening agents in nickel solutions. They also pointed out that although other aryl sulfonic acids gave reduced grain size, but no brightness, they also permitted the use of higher concentrations of brighteness such as cadmium and zinc salts, sodium formate, aldehydes, ketones, and amino polyaryl methanes, and enhance their action. An excess of these additions prevents deposition at low current densities and causes brittleness and poor adhesions. Bright nickel deposits have been found to contain 0.02 to 0.06% sulfur when deposited from a solution containing one of these organic sulfur compounds.

Kern and Fabian (18) tried deposition of nickel from a dithionate bath, but found it unsuitable due to separation of sulfur. Sodium sulfite was added to a nickel solution by Haring (171) who reported that dark deposits resulted at high current densities. Krzyzanowski and Gurewicz (412) have deposited a 1:9 copper-nickel alloy from a

both containing a copper chloride-thiourea complex, nickel sulfate and thiourea and acetic acid. Holland (168) has also reported the use of sodium thiosulfate in producing heavy nickel deposits. Young and Kenston (471), (449) have also formed several nickel-sulfur compounds by electrodeposition from a double nickel sulfate solution. Compounds containing from 27% sulfur and 73% nickel, to 8% sulfur and 87% nickel were formed.

Beutel and Kutzelnigg (75) found that nickel sulfide was deposited from nickel solutions when hydrosulfites were present. Lehner (400) reported that when nickel sulfate solutions were operated at high deposition velocities nickel bisulfate might form along with nickel peroxide.

Puri and Juneja (570) added a sulfur sol to a nickel plating solution and the deposit which resulted was black and spongy.

One of the most common sources of sulfur contamination is from rubber tank linings. According to True (619) the contamination may be caused by a black sulfur compound caused by the unreacted elemental sulfur in the rubber compound.

TIN

The Effects of Tin in Nickel Plating Solutions: Although tin is one of the more common elements, the possibility of its presence as an impurity in nickel plating solutions is probably slight. There may be tin contamination from bronzes which are nickel-plated, partivularly if they fall to the tank bottom, or become temporarily anodic, but nothing of this sort has been reported. In such cases, the effects due to zinc and copper which are usually present in higher concentration than tin in bronzes, would probably obscure any effects due to tin. Solanki and Singh (615) added trace amounts of stannous chloride ot nickel plating solutions and reported that the deposits were loose and powdery.

TUNGSTEN

The Effects of Tungsten in Nickel Plating Solutions: Tungsten has been added to nickel sulfate solutions so that a nickel-tungsten alloy could be electrodeposited. Although tungsten alone is deposited from aqueous solutions, when nickel is also present in the solution, the two can be co-deposited. Articles on the deposition of nickel-tungsten alloys have been written by Gol'tz and Kharlamov (41), Belyaev and Lipovetskaya (547), Silyarenko, Lyashina, and Masal'tseva (580), and Holt and Nielsen (628), (632).

ZI.3

The Effects of Zinc in Nickel Plating Solutions: The effects of zinc on nickel plating solutions has been well investigated. This fund of knowledge has been accumulated largely through investigations on the nickel plating of zinc-base die castings. Graham (163) undertook a study of the direct electrodeposition of nickel on zinc, and found that the presence of zinc in a nickel bath was not desirable. Thompson and Thomas (156) upon investigating the purity of nickel salts found that 0 to 1% zinc in nickel sulfate was not detrimental, but at 0.2% zinc the deposit which resulted from the use of these salts tended to be bright on the edges and also tended to pit. At 0.3% zinc in the nickel sulfate, the deposits tended toward bright edges and splotches of bright deposits. Setlik (307) also found that 0.115% zinc in a nickel plating solution retarded the rate of deposition, while 0.226% caused stains to form on the deposit. At 0.45% zinc the bath was useless. Anderson (364) also reported that the effect of more than 0.3 g./l. of zinc in a warm nickel solution was to brighten the deposit. Cracking and poor corrosion resistance developed reaching serious proportions at about 0.6g./l. of zinc. Anderson also observed that specimens plated from a solution containing 1 gm./l. of zinc in a sulfate-free solution, contained 0.2% zinc. Vozdvizlunskii and Makolin (454) investigated the causes of streaky deposits could be obtained in concentrations of zinc of 0.45 parts of zinc per 100 parts of nickel present. At a higher zinc content, between 0.45 and 0.65, streaky deposits of nickel developed, but the streaks could be removed by raising the bath temperature. Above 0.65 zinc the streaky deposits could not be avioded. They also noted that

the pH of the solution around the cathode increased with increase of zinc salts, reaching such a high value (6.07) that a colloidal precipitate of nickel hydroxide occurred. The positively charged particles of this colloid were then transported to the cathode surface and deposited as a dark layer. Meyer (607) has also written, "As little as 0.0067 oz./gal. of zinc will cause the nickel deposits at low current densities to be bright and somewhat more brittle. Higher concentrations of zinc will cause the nickel deposits to be very brittle and, for example, a concentration of 0.03 oz./gal.of zinc will cause the nickel deposits to be very brittle, particularly at the low current density range." According to Diggin (664) zinc causes dark plate to result from bright nickel solutions in low current density regions.

It is not accurately known when zinc was first added to nickel plating solutions as a brightener, but Proctor (68) in 1915 mentioned that zinc was being used for this purpose. Since this time zinc has been widely used in this manner.

One of the most common observations on the effect of zinc in nickel plating solutions is that it leads to dark or streaked deposits. Ter Doest (70), Field (131), Taylor (89), Thompson and Thomas (155), Haring (179), Voss (191), Wirshing (325), Rothersall (352), Snelling (360), Raub and Bihlmaier (392), and probably others, have all mentioned this effect.

Two observations have been made on the effect of zinc on the stress of resulting deposits. Vuillenmier (158) found that the presence of zinc caused an increase in the contraction of nickel deposits. Martin (657) has observed more recently, however, that in low pH Watts-type

solutions zinc does not materially affect stress, while at a pH of 5.5, zinc reduces stress. This later observation is in contradiction to Vuilleumier's observation which was also made on a high pH solution. Haring (179), (180) reported that the presence of zinc increased the throwing power of a nickel plating solution.

Zinc has also been a common ingredient in black nickel solutions, and in both cases its presence is deemed necessary. Many articles have been written on black nickel solutions which contained zinc. Some of these are by: Haas (112), (111), Lebolt (161), Watts (54), Blum (103), Hogaboom, Slattery, and Ham (117), (135), Harwell (182), and Barrows (288). According to Hogaboom, Slattery and Ham good black-nickel deposits usually contain 20 to 40% zinc, and therefore fall into the class of alloys. Other nickel-zinc alloys have been deposited, probably the first being done by Schoch and Hirsch (10). Investigations on the deposition of nickel-zinc alloys have also been carried on by Glasstone (226), De Mora (549), and Lustman (646). Izgaryshev and Ravikiovich (518) have also reported on the electrodeposition of a copper-zinc-nickel alloy.

To summarize the effects due to zinc in nickel plating solutions, the following effects can be considered as fairly acceptable facts. In the neighborhood of 0.007 oz./gal. of zinc is permissible as a brightener in dull-nickel plating solutions. Higher concentrations will yield brittle deposits, which may be either dark or streaked. In bright nickel solutions the effect of zinc in lower concentrations than 0.007 oz./gal. may become evident by darkening in the recesses, or at low current densities.

The Removal of Zinc from Nickel Plating Solutions: There are two general procedures for removing zinc contamination from a nickel plating solution. Probably the first mentioned was the precipitation method, or the Liscomb method (330). This method consists in first precipitating large quantities of ferric hydroxide in the nickel bath. If the iron content of the solution is not very high a quantity of an iron salt is usually added. The iron is also generally oxidized first either by aeration, or by the use of hydrogen peroxide. As the ferric hydroxide precipitate settles it adsorbs other metallic impurities and carries them out of solution. The solution is then filtered and the pH adjusted. The pH is ordinarily raised by adding nickel carbonate to the solution, and the use of heat aids in precipitating the iron. This treatment has been found by Anderson (364) to reduce the zinc concentration from 1 gm./l. to 0.0 25g./l. Wiesner (635) found that zinc was not appreciably removed from nickel sulfate solutions until a pH of 6.4 was reached, using ammonium or sodium hydroxide to raise the pH. At a pH of 6.6 and by heating the solution at 100°C for 2 hours, the zinc was completely removed. At this pH, however, considerable nickel was also precipitated. When the solution was buffered the treatment at a pH of 6.0 left about 300 mg./l. of zinc. Wiesner's investigations were made in the absence of iron as an impurity, so it is not certain what effect iron precipitating at the same time would have under these conditions. In most cases the drastic Liscomb method is not necessary to remove zinc. A typical procedure in precipitating zinc has been outlined by Stocker (617). This consists of heating the bath to 140°F., agitating the bath, and then raising the pH to 5.8 by adding freshly precipitated nickel carbonate as a slurry, slowly with much stirring, to the solution. About 2 to 4

pounds of nickel carbonate per 100 gallons of solution is normally required. A slurry of lime is then added slowly with vigorous stirring until the pH approaches 6.0 to 6.2, requiring about $\frac{1}{4}$ lb./100gal. of solution. About a half pint of 100 volume hydrogen peroxide is then added per 100 gals. of solution to oxidize the iron to the ferric state. The solution is then stirred throughly, allowed to settle, and finally filtered. This treatment is not only effective in removing zinc, but aluminum, iron, trivalent chromium, selenium, and cadmium. Costello (370), Savelli (519), Smith (538), Weisberg (496), and Wesley and Koehl (640) have also recommended this procedure, or one essentiall the same.

Zinc can also be removed from nickel plating solutions by electrolysis at a low current density. Mattacotti (484) has given a typical procedure of this type of purification.

"1. Heat bath to 135°-140°F.

2. Agitate continuously.

3. Fill tank with as large a cathode surface as possible.

4. Operate at a current density of 2 amperes/sq.ft, for copper and lead.

5. Other impurities operate at 5 amp./sq.ft.

6. Pass 2 ampere hours/gallon in case of copper and lead.

Pass 5 ampere hours/gallon in the case of zinc, etc."

Weisberg (496), (582), Anderson (364), and Diggin (664) have also recommended low current density electrolysis as a good method for removing zinc. This latter method is becoming more popular because it adapts itself better to continuous filtration methods.

SUSPENDED MATTER

The Effects of Suspended Matter in Nickel Plating Solutions: Suspended matter in nickel baths has long been a source of trouble. By suspended matter is meant particles of all sorts which are small enough, or light enough so that they are held in the solution for a period of time. Ferric hydroxide is probably the greatest single constituent of suspended matter. This precipitate is light and gelatinous and is easily suspended in the solution, aided by electrical and thermal currents. The term, "suspended matter" is generally considered to exclude colloidal particles, which are particles of such small size that they can not be removed by any normal filtration means. The effects of colloidal particles are similar to those caused by suspended matter, but there are also many effects so different that they will be discussed separately. Bancroft (4) was one of the first to recognize that oxides, hydroxides, and other basic salts could have a profound effect on nickel deposits. Blassett (33) also recognized that rough deposits could be formed when sediment settled on the work. Engemann (37) made a similar observation, and since ferric hydroxide was the principal kind of suspended matter, he proposed making the solution more acid. Barrows (46), on the other hand, suggested adding citric acid to prevent the formation of ferric hydroxide, and boric acid to prevent basic nickel salts from forming. Watts (57), Thompson (71), and Watters (72) also pointed out that slimes and suspended matter were a cause for rough and exfoliated deposits. In 1927 Hogaboom (229), (255) suggested that high purity nickel anodes be used to prevent the formation of sludge and floating particles. Hogaboom

also found that in some cases finely divided particles of metal were mechanically carried over from the anodes and deposited, causing rough deposits. Upthegrove and Baker (247) undertook a photomicrographic study of rough or nodulized nickel deposits, and found them to be due to suspended particles which had settled on the work and had been bridged by the deposit. Watts (248), Thomas and Blum (210), (211), Voss (191), Laban (401), Cosgrove (369), Cymboliste (410), (441), and Hothersall and Hammond (480), came forward with the suggestion that suspended particles were also a primary source of pitted and porous deposits. Ballay (341) showed that baths of a pH of 7 to 9 were unsatisfactory due to the formation of hydroxides and basic salts. He also found that boric acid caused a premature precipitation of hydroxides, and advocated the addition of citric, lactic or glycollic acid as a preventative of precipitation. Hothersall (352) felt that the basic matter in the cathode solution layer was the controlling factor in the properties of nickel deposits. Romanoff (358) also showed that the inclusion of basic particles in the deposit caused hard deposits, and Lemarchands and Debiesse (384) showed that nickel hydroxide also deposits with nickel. Liebrich (385) and MacNaughton (386) also pointed out that the cathode solution layer becomes alkaline during electrolysis, and that the basic salts which form these cause hardness, and may alter the normal crystal growth and orientation. More recently articles have been written by Cosgrove (408), Vozdvizhenskii and Makolin (434), Cymboliste and Salauze (442), Pinner and Berchert (489), Pohlschutter and Eggenberger (521), Wood (545), and Wernick (584) which repeat the observations already made, that suspended particles give rise to rough, porous, pitted, and even lus-

trous deposits. Thomas (638) has also pointed out that another source of suspended matter is fatty material which may get into the bath from contaminated work. Porter and Sward (205) have also pointed out that carbon particles from nickel anodes may be another type of suspended particle, and may lead to rough deposits. Pinner and Borchert (489) have also found that as much as 0.04% of loose nickel from anodes can cause rough deposits.

The Removal of Suspended Matter from Nickel Plating Solutions: The size of the particles of suspended material generally found in a nickel plating solution is large enough to allow their removal by filtration. The first type of filtration was simply carried out by filtering through cloth, but as time went on, better methods and materials were developed to improve the effectiveness of filtration and to speed up the process. The use of filter aids and activated carbon has advanced filtration techniques many-fold. The filters used for purifying electroplating solutions are generally constructed of stainless steel, bronze, or are rubberlined. Earlier filters were often of iron construction, but iron contamination sometimes resulted. Most filters consist essentially of a series of horizontal or vertical plates which contain filter cloth, which may also be coated with filter aid, activated carbon, and even nickel carbonate. The solution is forced through these plates, and thence into another tank, or into the same tank if continuous filtration is desired. Amberg (472) has given an excellent discussion of the use of filter-aids and has pointed out that they are used for four definite reasons: First, that they allow a higher filtration rate; second, longercycles

are obtained between filter cleaning; third, a variable degree of clarity can be obtained by using different grades of filter aid; fourth, through its use, the life of the filter cloth is extended. When filter-aid is used to precoat the filter-cloth, it is mixed with water and passed through the successive cloths, until each is coated with a layer of the filter aid. Most filter-aids are diatomaceous earths. Meyer (485) has written an article on the methods of mixing filter-aids with plating solutions which is very instructive.

For a discussion of filter pumps, articles by Weisberg and Greenwald (362), Pace (390), (425), and Belke (501) are very instructive. Other articles on the general subject of filtration and purification which are very good are: Hobbs (48), Shepard (187), Watts (248), Laban (276), Jevell (519), Meyer (529), Smith (538), (539), Green (556), (599), Wernick and Silman (585), Wynne-Williams (589), (590), Helbig (603), Stocker (618), Rankin (637), and Case (663).

Green (599) has also issued a note of caution in pointing out that rough nickel deposits can result from filter-aid and activated carbon passing into the solution. Weisberg (582) has also pointed out that once activated carbon gets into a solution, it is difficult to remove.

ORGANIC SUBSTANCES

The Effect of Organic Substances in Nickel Plating Solutions: Citrates have been one of the most common of organic substances in nickel plating solutions. They were first used for keeping iron in solution by forming a soluble complexit. Barrows (46) was probably the first to make this recommendation. Mathers (81) found, however, that the addition of citrates increased the amount of iron codeposited with the nickel. Since the codeposition of iron was not considered to be as bad as the effects of sludge in the tank, the addition still received some popularity. Articles were successively written by Haas (94), Later (99), Werner (145), Mathers (257), Ballay (341), (343), Raub (391), Raub and Walter (393), Mathers (420), and Krause (562) recommending citrates as an addition for keeping iron in solution. Another popular use of citrates and citric acid was in the electroplating of zinc and aluminum materials. Weber (74), Nelson (108), Proctor (124), Field (151), Ledin (151), Thompson (212), Foss (253), Werner (691), (623) and Trentmann (399) have found citrates of value in plating these materials. Haring (179) also observed that sodium citrate additions greatly increased the throwing power of a nickel solution. Martin (657) found that citrates also decreased the stress usually found in nickel deposits when used in solutions at both low and high pH. Another common use of citrates is their buffer action. Cotton (409), and Meyer (454), have found them to be excellent buffers, and Pollack (651) has made a similar observation in regard to citric acid. Citrates have also been noted by Werner (145) to make deposits tougher.

Formates are also used in nickel plating solutions. Kersten and Young (418) used nickel formate in depositing nickel-iron alloys. The most common use of formates today is as a constituend ot bright nickel baths. Weisberg (542),(582), (583) has investigated the use of both nickel and sodium formate in nickel baths. The action of both compounds is similar, they tend to make the deposit harder, act as buffers, and make a contribution to the brightness of the bath. The formate is usually used along with formaldehydr in the Weisberg baths. Martin (657) investigated causes of stress in nickel plating solutions and found that the addition of formate decreased stress in both low and high pH solutions.

Acetates have been used as buffers in nickel plating solutions. O'Sullivan (282), Watts (310) and Esin and Leshkarev (505) have found acetates to be satisfactory buffers. Acetic acid has also been added as a buffer by Cotton (409) and Krause (562) and Haas (94). Martin found that as in the case of formates, acetates decreased the stress in nickel deposits.

Tartrates have been common addition agents to nickel plating solutions. Haas (132), (133), found Rochelle salts to be a valuable constituent in black nickel solutions, and Mathers, Webb, and Schaff (354) have used Rochelle salts with ethanolamine in alkaline plating baths for nickeland cobalt. Along with citrates, tartrates have been used to keep iron soluble in solution. Raub (391) found that this addition prevented the accumulation of free iron.

Lactates are similar in action to the compounds already mentioned, although their use has been more recent. Nichols (334) has found

that lactates retard the deposition of nickel on zinc by immersion, and that they do not produce the yellow colored deposits which citrates tend to form.

Organic Acids particularly the acids of the salts already mentioned have frequently been added to nickel plating solutions. Haas (94) added benzoic, tartaric, acetic, and succinic acids to nickel plating solutions and found that they gave large polarization values, so were therefore unsuitable addition agents. Citric acid was found to give satisfactory deposits. Ballay (341), (343), investigated glycolic, lactic, and citric acids for preventing the precipitation of iron and found all to be effective, particularly citric acid. Malic and tartaric acid were found to be without action. Raub (391), however, found that citric and tartaric acid additions yielded good deposits, but lactic acid caused poor deposits. Krause (562) has found that acetic, citric, and tartaric acids were all effective in preventing the precipitation of iron, manganese, and chromium hydroxides. Cotton (409) investigated acetic, citric, lactic, succinic, formic, and tartaric acids as buffers, and Pollack (651) found tartaric, glyoxalic, and glycolic acids to be excellent buffers, and that baths containing these three acids could be used to plate light metals. Pollack found the deposits from such baths to be superior, in appearance and adherence, to those from baths containing citric acid. Formic acid has been used primarily as a brightener, and as such has been investigated by Weisberg (583) and Raub and Wittum (572). The latter authors found that formic acid caused a weak increase in brightness, but had no effect on the mechanical properties of the deposit. In

nickel-cobalt solutions formic acid was found to increase the reflectivity of the deposit markedly. Raub and Wittum (572) also investigated pyroacemic and levulinic acids, but found them to have little effect on either the brightness or physical properties of the deposit. Flasunev and Schlötter (445) have pointed out an important fact regarding the use of these compounds which form soluble complexes in solution, and that is that undecomposed complexes enter into the deposit lattices, and may therefore affect the pjysical properties of the deposit.

Liniek (605) investigated organic additions agents, and found that the addition of benzaylacetic, diphenylacetic, phenylaceturic, benzenesulforric, toluic, and tropic acids produced unsatisfactory results as brightening agents. When Raub and Wittum (572) investigated aromatic acids they found that benzoic and salicylic acids had no effect on the brightness of nickel. Acetylsalicylic acid, however, produced milky white to brilliant deposits in concentrations from 0.1 to 0.2 g./l., while other acids which they investigated, even in tinfold concentration produced no effects. The mechanical properties of deposits obtained from solutions containing acetylsalicylic acid were good. Phthalic acid and phenolphthalein produced no noticeable effect. When tannin was added to a nickel bath, it was only slightly soluble and no distinct effects were observed for the small quantities which were soluble.

Alcohols have been added to nickel baths, but even in large amounts they have little effect on nickel deposition. Raub and Wittum (572)

have made this observation in regard to methyl and ethyl alcohols. When a bath was saturated with iso-amyl alcohol, the deposits were milky in color, but the physical properties were altered only slightly. Of the alcohols, the tri-hydroxy alcohol, glycerin has been the most frequent addition to nickel plating solutions. In 1921 Watts (144) added a mixture of fluoborate and glycerin to a nickel bath and noted that the resulting deposit was remarkably smooth. Shepard (186), (209) used a lead and glycerin combination as a brightener, and found that both the lead acetate and the glycerin contributed to the brightness of the deposit. Schmeh (260), and Werner (291) have found glycerin to be a valuable addition to nickel baths used for plating aluminum. Mathers and Guest (565) have found that glycerin reduced the throwing power of a nickel plating solution when operated at ghih current densities. Raub and Wittum (572) added 50 cc./l. of glycerin to nickel plating solutions and found that a much brighter but milky deposit was formed. Deposits with a thickness of about 0.00024 in. had a reflectivity almost as great as buffed nickel. The deposits had, however, a golden coleration, and as the thickness increased, the coatings became more matte, and less reflective. When the glycerin was added in amount exceeding 50 cc./l. the deposits became non-uniformly cloudy, and matte. Raub and Wittum also noted that hydrogen has a greater tendency to adhere to the cathodes in solutions containing glycerin. In small concentrations glycerin did not produce any effects on the deposit. Stout (432) has pointed out that polyhydric alcohols, particularly the glycols, are being increasingly used as nickel brighteners.

Aldehydes and Ketones have been added to nickel baths, and of

these formaldehyde has been the most investigated. Weisberg (542) has found formaldehyde to contribute very definitely to the brightness of nickel-cobalt plating solutions. Nickel deposits can be produced with this addition which produce reflectivities closely approaching buffed nickel deposits. Raub and Wittum (572) have found that when formaldehyde is used as a brightener, the brightness is dependent only on the surface condition of the base plate. In thick deposits, even a matte base plate will result in brilliant deposits. The deposits from solutions containing formaldehyde show very definite layering. Photomicrographs illustrating this layering phenomenon in formaldehyde-containing baths and other bright nickel baths are to be found in papers by Raub and Wittum (572) and by Young (592). Raub and Wittum (572) also observed that the brilliant deposits from solutions containing formaldehydr tended to be brittle, and that cracks might form on bending, but only in spots subjected to the greatest deformation, and exfoliation parallel to layering is not found. Brittleness was found to increase with increase in thickness of the deposit, but no noticeable decrease in adhesion was found. Apparently certin organic materials are codeposited with nickel in solutions containing formaldehyde, since an unpleasant odor is noticed when the deposit is heated. Raub and Wittum attribute this pehnomenon to the polymerization of formaldehyde by which colloidal matter forms. They also noted that formaldehyde increased the polarization decidedly for nickel deposition. In still baths at low current densities, the presence of formaldehyde was found to lead to brittle and streaked deposits. When the solution was agitated, however, the polarization at higher current densities was less than in still solutions. Raub

and Wittum (572) felt that the solution they investigated had its best properties with a formaldehyde content of from 1 to 14 oz./gal. These authors also investigated acetaldehyde and propylaldehyde additions. With acetaldehyde the effect was similar to formaldehyde, only stronger. The mechanical properties of the deposit were impaired however, and it was under high tension, and covered with hair-like cracks. propyladehyde in amounts up to 50 cc. per liter had no effect on the brightness of the deposit, and an increase in hardness and a decrease in ductility occurred. The addition of acetone at 10cc./l. resulted in milky bright to bright deposits, but the deposits tended to exfoliate in scales, even in the bath. Heavier coatings from acetone containing solutions were gray to black gray. Linick (605) added benzylacetone to a nickel bath, but an unsatisfactory deposit resulted.

Carbohydrates have been added to nickel plating baths, primarily to find out if they have brightening properties. Gelatin has been one of the more common of these additions. It is probably true that the effects of gelatin glue, etc. Are due primarily to their colloidal properties, and as such they have been widely studied addition agents to nickel baths. In 1911, Proctor (38) recommended the addition of a small amount of gelatine or transparent white glue to produce bright nickel deposits, providing the deposit was not to be very thick. Vuilleumier (145) then investigated the effect gelatin had on the contraction of nickel deppsits, and found the effect to decrease the contraction. Frölich (176), and Frölich and Clark (200) also investigated gelatin additions and proposed that the effect of gelatin at the cathode surface was to accumulate and prevent circulation of the electrolyte, or actually to dilute the electrolyte at the cathode surface.

In high pH nickel solutions Frolich found carbon in the deposits, which is in disagreement with the expected migration of gelatin particles to the anode at a pH greater than 4.7. Ballay (344) found bright deposits to result after the addition of gelatin, and reasoned that solutions which contained colloids which pre-exist in the bath give more uniform results over a wider plating range than those which form in the cathode zone. According to an anonymous German author (565) gelatine is an effective brightener in concentrations as low as 2.5 mg./l. Lewis (203) found that the addition of gelatin to a single nickel salt bath which exhibited normal throwing power of 32% was reduced to 17%, and the cathode efficiency was also reduced to 88.5%. Cahour (407) found that gelatine increased the hardness of nickel deposits up to a certain concentration, and then began to decrease it. Hothersall and Gardam (479), and Mattaeotti (527) have also found gelatin to yield brittle and hard deposits. Fischer (662) found that organic compounds like gelatin first showed signs of concentration polarization and high resistance at the cathode at a concentration of 10g./l. or above. A more recent theory of the action of gelatin has been proposed by Glasunov and Dreseher (414) who believe that the addition of gelatin and other colloidal materials causes a reduction in the numerical value of the crystallization velocity, which is not dependent on a decrease in viscosity. Kochanovskii (450) has proposed that nickel ions become inactivated by micelles of lyophilic colloids like gelatin, and that the inactivation is greater the more lyophilic the colloid is. He has not tried to adapt this theory to the effect of gelatin in nickel plating solutions, but it may be a plausible theory. Glue exhibits effects similar to

gelatin when present in nickel plating solutions. Canning (294), Liscomb (330)9 Hothersall and Gardam (447), Shcherbakov, Loshkarev, and Koshkarev (535), and Matta cotti (527) have pointed out that glue, varnish, resin, and sap from wood can severely contaminate a nickel bath. According to Liscomb (330), if glue is present the bath will froth, and the deposits are bright, streaked, and brittle. Gelatin and glue are really proteins, and other proteins such as albumin, casein, and peptone act quite similarly. Raub and Wittum (572) have pointed out that all protein substances do not act alike. Casein, which is only soluble in small quantities was found to be very detrimental to nickel solutions. They observed that scum was formed on sur-face of the cathode, and as a result the base metal was only incompletely covered, and the adherence was very poor. They also found that the cathode potential fluctuated quite strongly after casein was added, and found that a usable cathode potential-current density curve was impossible to make. Raub and Wittum also recorded similar effects for albumin, peptone and glue. Additions as small as 0.01-0.05 g./l. were formed however, to allow the deposition of plates of high spectral reflectivity, which approached that of buffed nickel. The ductility was found to be very poor in these deposits, and on bending the specimens cracked in spots of greatest tension. The brilliance of the deposit was found to increase with the protein concentration and with the thickness of the deposit. At higher protein content they observed the deposits to be discolored. Raub and Wittum also observed that with small additions of protein to an agitated bath, a strong increase in cathode potential was observed, but in a still

bath, only a small increase was observed. They explained this phenomenon as being due to the fact that with a small protein concentration in a still bath, the protein at thecathode is quickly depleted, but when the bath is agitated, the protein at the cathode layer is being continuously replenished. When the temperature was increased to 55°C. in a bath containing peptone, the brittleness was only slightly reduced. Mandi (304) has investigated the effect of albumin on the deposition potentials of metals, and Ballay (500), (344) has discussed the brightening action of gelatin, starch, dextrin, gum arabic, agar agar, egg albumin and casein. Robinson (87) reported in 1916 that 1 oz./gal. of yellow dextrin made an exed lent brightener solution. This solution was ad ed to the bath as one fluid oz./300 gallons of solution. Gum arabic and gum tragacanth have been added as brighteners to nickel plating solutions by Mills (100), Underwood (263), and Sizeleve (462). Sizeleve found that an excess of gum arabic caused dark streaky deposits, and where the streaks occurred, little or no nickel was found.

Sugars are other carbohydrates which have been investigated as brightening agents in nickel plating solutions. The use of glucose has been mentioned by Blum (112), and mannitol and sucrose by Selmki and Singh (615). Stout (432) has also listed sucrose, corn sugar, and malt syrup as brighteners. He also found that glucose added in concentrations of 1½ to 2 oz./gal. yielded improved nickel deposits over those from ordinary gray-nickel baths. Raub and Wittum (572) investigated dextrose, lactose, and maltose additions and in the case of dextrose found that the deposits were only milky bright at best. Lactose in concentrations of 4 oz./gal. had little influence on the reflec-

tivity of the coatings, and above 4 oz./gal. of lactose the coatings again became matte. Neither dextrose nor lactose were found to materially affect the mechanical properties of the deposit in concentrations between 2.5 and 5.4 oz./gal. Maltose additions resulted in streaked deposits at a concentration of 0.67 oz./gal. which tended to be brilliant at times. The mechanical properties of the deposit were poorer thatn these from solutions containing dextrose. Methyl cellulose, another carbohydrate, was also investigated by Raub and Wittum and at a concentration of 0.02 oz./gal. the deposits were not affected. The potential for nickel deposition was displaced, however, about 20 to 30 millivolts toward the non-noble potential.

Organic Nitrogen compounds have been systematically investigated by Raub and Wittum (572) to determine their effects in nickel plating solutions. Formamide was found to increase the brightness of deposits, but their mechanical properties were seriously impaired. At a concentration of 0.067 oz./gal. of formamide the deposits were non-uniform, matte bright to brilliant deposits, which showed good adhesion and ductility on bending, when the coatings were not very thick. In a solution containing 0.13 oz./gal. of formamide, the deposits became exfoliated in the solution as soon as their thickness exceeded 0.00016 inches. Urea additions accentuated pitting in a nickel solution, due to increased adhesion of hydrogen bubbles to the cathode. The brightness of the deposit was not increased by urea additions, and the polarization of nickel deposition was increased. Methyl urea additions yielded matte, hard, and brittle deposits. Uric acid additions in concentrations of 0.005 oz./gal. caused marked increase in the brightness

of the deposit. The adhesion of the deposit was good, but due to internal stress, the ductility was poor. Thicker coatings were found to be full of fine cracks. When the concentration of uric acid was increased to 0.13 oz./gal. the deposits were brilliant, but exfoliated in the solution. As the uric acid concentration increased, the amount of hydrogen evolution increased, even at low current densities. Caffeine which is a compound derived from uric acid was found to markedly disturb nickel deposition even in concentrations of 0.001 oz./gal.

Semi-carbazide, another urea derivative, produced less marked effects than urea at a concentration of 0.003 oz./gal. as regards spectral reflectivity. The mechanical properties were but little influenced. As more semi-carbazide was added, the deposit again became matte. Ethyl carbamate, or urethone, had a powerful brightening effect and with additions of 0.026 oz./gal. brilliant deposits were obtained after 30 minutes of plating. Polarization and hardness were also increased by urethane additions. Oxamide additions were found to cause a strong evolution of hydrogen, and this evolution increased with oxamide concentration and current density. Thus only low current densities were permissible with this addition, and the deposit produced showed only a weak increase in reflectivity but no marked change in mechanical properties. At high current densities the deposit was dark and spongy. Glycocoll was tested as an example of an amino acid and at a concentration of 0.3 g./l. there was no noticeable effect on the deposit. Guanidine sulfate was also found to produce spongy and pulverant deposits, and was harmful in all concentrations. Hexamethylenetetramine was apparently similar in its effects to formaldehyde. Since it is formed by the combination of formalde-

hyde with ammonia, Raub and Wittum reasoned that the hexamethylenetetramine broke down into these compounds in the bath. Another amino acid, phenylalanine, was found by Linick (605) to produce an excellent brightening effect when added to nickel solutions. In 1920 Aliverti (119) added strychnine to a nickel bath and found that it decreased the contraction of the deposit.

Raub and Wittum (572) also investigated certain aromatic nitrogen compounds. They found no appreciable effect on increasing brightness of nickel deposits by aromatic amines, but a marked increase in polarization of nickel deposition was noted. Methyl red, a sulfate-free azo dye did not increase brightness, but methylene blue, which contains sulfur, did increase the brightness of the deposits. Concentrations of 0.02 to 0.1 g./l. of methylene blue produced brilliant deposits which closely approached buffed nickel in reflectivity. The deposits were found to be highly stressed however, and brittle, and usually were observed to exfoliate in the solution. Saccharin was also found to be a strong brightener, and the best results were obtained using 0.1 to 0.2 g./l.. With higher concentrations the brilliance began to diminish, with coincident impairment of mechanical properties.

<u>Sulfur-containing aromatic compounds</u> were also found by Raub and Wittum (572) to have marked effects on nickel deposition. Ethyl mercaptan and allyl mustard oil were added, but their obnoxious odors limited their use. In concentrations up to 6.5 c.c./l. of ethyl mercaptan, and up to 10 c.c./l. of allyl mustard oil, the deposit was distinctly brightened, and no loss in mechanical properties was no-

tioed. Thiourea was found to be a strong brightening agent, and the deposits produced exceeded even those from formaldehyde-containing solutions. An addition of only 0.15 g./l. was sufficient to produce deposits as reflective as buffed nickel. The deposits became increasingly brilliant with thickness, but the cohesion of thick deposits was weak. When the deposits were bent, the nickel not only cracked at the bends, but exfoliated in thin sheets. When deposits from thiourea-containing baths were heated small bubbles of gas appeared all over the surface which reacted distinctly alkaline, therefore, indicating an amine. When the deposit was dissolved in hydrochloric acid, the gas evolved contained hydrogen sulfide and traces of a gas with an odor like methyl sulfide which was not absorbed by alkaline or iodide solutions. Oxidation and polymerization products of thiourea were also noted to form slimy and partly fibrous material on the anodes. The cathode potential-current density curve was strongly displaced to the negative side by thiourea additions. At high current densities, the potential measurements were uncertain and strong fluctuations occurred which led gradually to lower potential values. Cystine, a sulfur bearing amino acid had no distinct effect on nickel deposition at a concentration of 0.15 g./l.

Proctor (68) mentioned in 1915 that ethyl sulfate was a good brightener, and Riedel (85) recommended the use of sodium ethyl sulfate.

Sulphonic Acids constitute a great majority of the brighteners added to modern bright nickel baths. Besides being added as brighteners, certain of these acids and their salts have been used as wetting agents. Eckardt (373) in 1935 recommended sodium phenolsulfonate as a wetting agent. Blount (474) found that an alkyl aromatic sulfon-

ate caused a whiter, brighter plate to form, which was also softer, finergrained, and easier to buff. The sulfonate also prevented the adherence of hydrogen bubbles to the cathode. Too high a concentration of this wetting was formed, however, to cause pitting and bare spots. Stocker (540) has also pointed out that wetting agents as a whole make the plate more susceptible to grease contamination. Since 1938 some excellent articles have been written on the use of wetting agents in nickel plating solutions in particular, and all plating solutions in general. Some of these are by: The Newark Branch of the American Electroplater's Socity (487) Young (546), Hartshorn (558), Samartsw (574),Crowe (594), Davis, Wolfe, and France (595), Planner (635), McFarlane (660), and Silman (661).

Stout (432) mentioned in 1936 that aromatic sulfonic acid derivatives were finding increasing use as brighteners in nickel solutions. Springer (464) made the same observation a year later and Hothersall and Gardam (516) in 1939 investigated the structure and properties of nickel deposited from baths containing 5 g./l. of nickel naphthalene trisulfonate. Another bath these authors investigated was one containing 30 g./l. of sodium iso-propyl naphthalene sulfonate. Young (592) in his study of bright nickel processes mentioned the use of naphthalene tri-sulfonic acids, sulfonated oleo resin, and benzene or o-toluene sulfonamide. Maurer (606) also found that sulfonated benzene or naphthaline derivatives made deposits more dustile. Hendricks (627) made a similar observation, pointing out that when brightmers such as zinc, cadmium or the poly aryl methane dyes are used alone, a hard brittle nickel deposit is obtained.

If an aryl sulfonic is used in conjunction with these brighteners a bright and ductile deposit is obtained. Pinner, Soderberg, and Baker (609) listed polysulfonic acids, aryl sulfonamides, and aryl sulfonimides as brighteners in nickel baths, and listed other aryl sulfonic acids as grain-refiners, but not brighteners. These latter sulfonic acids permit, however, larger amounts of brighteners such as the salts of cadmium and zinc, formates, aldehydes, ketones, and amino polyaryl methanes to be used and even enhance their action. Stepanov and Lyashchenko (616) also found sulfonated naphthalenes to be valuable brighteners, and Pollack (636) made the same judgement concerning sodium phenolsulfaonat. Linick (605) found no brightening action from the additions of potassium phenyl sulfonate, benzenesulforic acid, and sodium benzene sulfonate.

Rumb and Wittum (572) have made the most complete and extensive investigation of aromatic and heterocyclic sulfonic acids, and their findings have yielded valuable information. Although they found few generalities regarding these compounds, they did find that deposits from solutions containing sulfonic acids always evolved hydrogen sulfide gas when dissolved in hydrochloric acid. They also found that the amount of sulfur codeposited was dependent on the current density and the type of sulfonic acid. The sulfide form of sulfur which was detected in the deposits was found to be from the decompesition of the sulfonic acids, and the brightnessof the deposit was independent of the sulfur concentration. The nickel being deposited was also found to catalyze the break-down of these compounds and when the bath was operated for some time the odor of sulfonic acid-free compounds could

be detected in the aromatic sulfonic acids were all found to displace potential-current density curves in a negative direction.

Potassium phenolsulfonate was investigated and found to be ineffective in concentrations up to 8.6 g./l., as a brightener, and the deposits were brittle and covered with fine cracks. Alpha and beta naphthalene sulfonic acids were found to be powerful brighteners. The alpha acid as the more effective of the two, but both produced deposits approaching buffed nickel in reflectivity. Mechanical properties of deposit were not impaired as long as the concentration of the naphthalene sulfanic acids was kept low. Concentrations of 0.2 to 0.6 g./l. were satisfactory, while higher concentrations led to exfoliated deposits. Naohthalene-1,3,6, sodium trisulfonate was investigated as a typical tri-sulfonate, but the deposits were milky bright at best. The adhesion and ductility of the deposit were only slightly diminished. Anthracene-1 sulfonic acid in the small concentration which was soluble in the bath and phenanthrene 3-sulfonic acid at a concentration of 4 g./l. were uneffective.

The 1- and 2- monosulfonic acids of dibutylnaphthalene produced excessive trubidity in the bath at a concentration of 3 g./l. Bright deposits were, however, obtained, which were poorly adherent, and poorly ductile. When the concentration wasreduced to 2 g./l. the degree of brightness remained, nd the mechanical properties of the deposit were generally better. Another slightly solutlb compound, benzyl napthalene sulfonate, was also tried, but yielded only milky bright deposits. Of several unknown poly-substituted napthalene sulfonic acids tried, only one was satisfactory as a brightener. This com-

pound was a strong brightener even in concentrations of 0.1 g./l., but it greatly increased the polarization of nickel deposition. The addition of 0.1 to 0.5 g./l. of 2 naphthol-6-sodium sulfonate, 2-naphthol sodium disulfonate, 3-naphthol-3,6,8-sodium trisulfonate, and 2-naphthol 3,6,8-sodium trisulfonate produced deposits which were very nearly as bright as buffed nickel. The deposits however, were internally stressed, were covered with hair-like cracks, and were poorly adherent to the base metal. Bad cases of pitting were also observed through the use of these compounds.

The sodium salts of anthraquinone and dioxyanthraquinone sulfonic acid were found by Raub and Wittum to be strong depolarizers, and therefore led to dark and poorly adherent deposits. The 2-5,2-6, 2-7, and 1-4, naphtholamine sulfonic acids were added in concentrations up to 2 g./l., but the increase in brightness which they produced was slight in comparison with that produced by the naphthalene and naphthol sulfonic acids. The hardness was markedly increased, but the ductility was only slightly decreased. 2-amine-5 naphthol-7 sulfonic acid, methyl orange, and congo red were found to be unsatisfactory brighteners. Quinoline-8-sulfonic acid in concentrationd of from 1 to 6 g./l. gave matly bright to brilliant coatings with fairly good mechanical properties. When the concentration exceeded 6 g/l. the deposits became dull and discolored, and the mechanical properties were also impaired.

Heterocyclic compounds were also investigated by Raub and Wittum (572), particularly fuffurol, pyridine, orthohydroxyquinoline, and quinine. Furfurol was found to increase the brilliance of deposits

quite strongly. In concentrations between 0.2 and 0.6 g./l. the reflectivities of deposits were only about 10% lower than buffed nickel, and the brightness was independent of thickness. A few drops of pyridine per liter resulted in loss of adhesion of the deposit to the base metal, and the coating was not noticeably brightened. Orthohydroxyquinoline was even more effective as a brightener than furfurol, and as long as the concentration was not very high, the properties of the deposit were not appreciably lessened. The optimim concentration of saturated orthohydroxyperinoline solution was about 20 cc./liter of nickel solution. Quinine acted as a depolarizer, and at higher concentrations prevented nickel deposition.

Besides the organic materials mentioned there are several other organics which have been discussed. One of these is living organic matter. Ollard (335) recorded that three types of micro-organisms have been found in nickel baths, but their effects are not Brown. Ollard (219) also found that Penicillium glucinium, a micro-organism, has been found growing in nickel plating solutions. Darlay (371) has also pointed out that a bath may develop surface mold by fermentation of organic matter from water or brighteners in the bath. Stopping-off materials have been another source of organic contamination in nickel baths, as pointed out by Macnaughton and Hothersall (281) and Waite (666). Other sources of organic contamination mentioned by Waite are filter cloths, suede bags, and tank linings.

Organic materials, regardless of their type have long been known to cause ill effects in electroplating solutions. The effects attributed to organic contamination were: brittleness, hardness, pitting, peeling, and dark or streaked deposits. Besides the articles mentioned

previously, articles by the following authors also mentioned these effects: Mathers (139), Madsin (182), Porter and Sward (205), Ollard (219), Shepard (220), Darlay (345), Snelling (360), Shaherbakov, Leshkarev, and Leshkarev (576), Stocker (618), Meyer (529), Mattaeotti (527), Case (665), Diggin (665), Waite (666), and others.

The Removal of Organic Substances from Nickel Plating Solutions: There have been several methods suggested for removing organic contamination from nickel plating solutions, and all of them are more or less effective. Probably the first method suggested was the chlorination method of Madsen (182). In this method chlorine gas is bubbled through the solution to destroy the organic matter. More recently Hothersall (352) recommended the chlorine treatment again followed by the addition of Kieselgahr and filtration. This type of treatment is obviously a difficult one to perform since chlorine is a poisonous gas. A second method which has received more popularity is the hydrogen peroxide oxidation method. Hydrogen peroxide is an effective oxidizing agent and will destroy most organic matter. This method was suggested in 1925 and 1926 by Porter and Sward (205) and Dobbs (215), although the method was evidently in use before this time. A typical procedure for hydrogen peroxide treatment for removal of organic contamination has been given by Mattaeotti (484). This consists of (1) Heating the bath to 140°F. (2) Adding while stirring $\frac{1}{2}$ gal. to 2 gal. of 100 volume hydrogen peroxide. The variation in amount allows for the extent of organic contamination. (3) Allowing the solution to stand warm overnight with agitation. If the throwing power of the bath is decreased, Mattaeotti recommends that the temperature be raised to expel any excess peroxide.

Another oxidation method which has been used to some extent is the potassium permanganate treatment. This treatmtne was suggested by Shepard (220) in 1926. Shepard preferred potassium permanganate to hydrogen peroxide since the peroxide tended to flocculate colloidal ferric hydroxide. Hothersall and Gardam (447), and Thomas (638) have also found potassium permanganate to be very effective. There is one factor in favor of the use of permanganate, namely as long as organic matter remains in the bath, the permanganate will be decolorized. As soon as the organic matter is destroyed, the purple permanganate will turn to brown manganese dioxide, thereby indicating when the reaction is complete. The effects of manganese are not well known in nickel plating solutions, but one argument against the use of permanganate is that it adds manganese as a metallic contaminant to the bath. A method of treating contami ated bath with potassium permanganate has been given by Weisberg (541) who suggested that the pH of the solution be brought to a value of 2, heated and then 3 lbs. of potassium permanganate per 1000 liters of solution be added. The solution is then allowed to stand to allow the permangsnate to react, and then allowed to cool. The pH is then raised by the addition of nickel carbonate until the excess permanganate precipitates out as manganese dioxide. The precipitate is then filtered off, and the pH adjusted.

Another method was suggested by Darlay (371) which appears to be unique. He suggested that organic matter be removed by filtering the bath and then treating it with salicylic acid or mercuric chloride.

Probably the most widely used effective method for the removal of

organic matter is by the use activated carbon. Articles by Helbig (478), (603) have fully described the use of activated carbon. This material is highly porous, and possesses extremely large surface area. The particles of this material exhibit tremendous adsorptive powers for most organic materials found in nickel plating baths. Helbig recommended that the carbon be precoated onto the filter by the slurry technique, using sulficient carbon to fill about three-quarters of the available filter cake space. He also found filter-aids as effective partners to activated carbon in aiding filtration both in speed and efficiency. The circulation of solution through the filter was recommended to not exceed 25 gals./hour for each square foot of effective filter area. Activated carbon has also been added directly to the contaminated solution and thoroughly agitated. Such a procedure has been recommended by Mattacotti (484), Smith (538), Stocker (617), (618) as being more effective, providing steps are taken to effect complete removal of carbon from this solution. To do this Stocker (617) recommended that a separate treating tank be used, so that there would be no chance of carbon remaining in the solution. When a solution is treated in this manner it is usually necessary to precoat the filter with filter aid to make certain that the carbon does not pass through the filter. Apparently pre-coating the filter with carbon is the preferable method. It may require a longer purification period than the direct solution addition method, but it eliminates danger from carbon contamination. An article by Helbig (603) very completely covers the use of this material in purifying plating solutions, and is recommended to anyone for removing organic contamination in nickel baths.

Other excellent articles on the activated carbon method of purification are by: Jovell (519), Mattacotti (527), Meyer (529), Smith (528), Weisberg (541), Shcherbakov, Loshkarev, and Loshkarev (576), Weisberg (582), Stocker (617), (618), Case (663), and Diggin (664).

A method of removing organic matter has been proposed by Weisberg (468) which is based on high current density electrolysis. Not all organic matter can be removed in this manner, but it is effective on some types of contamination. The organic material which are destroyed have not been well-classified. Mattacotti (484) pointed out that the loss of metal by high-current density electrolysis could be lessened by lowering the pH to 2. The electrolysis is carried out between 2 and 150 amps./sq.ft. depending on the type and degree of contamination. Due to the uncertainty as to whether the contamination will be removed, and the cost due to loss of nickel, this method has not been extensively adopted.

In general the carbon treatment is probably the best method where the quipment is available. Continuous purification is coming into more extensive use, and activated carbon also has the advantage of lending itself to such a practice. Diggin (664) has pointed out that in cases where activated carbon does not effectively remove organic contamination, treatment with activated clay is often effective. The peroxide method does have an advantage in that the procedure also removes iron and small amounts of other metallic impurities along with it. In this connection, the purification method of Liscomb (330) should also be mentioned since the large amount of ferric hydroxide which is formed by this method also removes some organic contamination.

Effects of Colloidal Matter in Nickel Plating Solutions: Colloidal matter of specific compositions have already been discussed. However, a discussion of the general effects of colloids should be made since they have great bearing on the electrodeposition of metals. the theory of colloid action on electrodeposition has gone through an interesting develoopment, and it is difficult to make a decision on any particular theory since the cathode layer where the fundamental reactions of electrodeposition take place is extremely thin. One of the earlier concepts was that of Betts (5) who felt that any action a colloidal material had on electrodeposition was due solely to some chemical reaction. It is true that most colloidal substances have individual properties, but there are a geat number of effects which appear to be common. This sort of a phenomenon would be difficult to explain from the concept of Betts. One of the general effects attributed to colloids is that they refine grain structures of electrodeposits. Blum (112) and Blum and Rawdon (160) recognized this latter fact and attributed the effect of colloids such as glue and gelatin to the increase in vixcosity of the solution, thereby hindering convertion and diffusion, and thus reducing the effective metal ion concentration at the cathode. They also felt that if the colloid migrated to the cathode and accumulated, it would probably be adserbed by the deposit, and thus alter the arrangement of the crystals of the deposit, and their growth. Blum and Rawdon attributed the specific effects of different colloids to differences in the degree of their adsorption by the deposited metal.

Another early observation was made by Whitcomb (126) who pointed out that colloids could carry electrical charges, and might therefore mi-

grate to the electrode of opposite charge from their own. He also felt that colloids tended to prevent the formation of tank sediment. Kohlschutter (150) also made the observation that colloidal absorption films might cause overvoltage. One of the most complete investigations of the role of colloids in electrodeposition was made by Sand (141), (154) who investigated the following: their colloidal nature and inclusion in the deposited metal; their gold-number and absorption relative to their effectiveness; their action after adsorption; microstructure of deposits; equilibrium potential; transfer resistance and polarization; over-voltage effects; mechanism producing the final structure of electrolytic metal deposits; the importance of colloidal substances accidentally present or incidentally produced; colloids at the anode; and colloids in applied metal deposition.

Solution composition has an effect on the form of electrolytic deposits, and Hughes (166) has found that the presence of colloids affect the influence of solution composition. Macnaughton and Hothersall (245) in a discussion on the causes of pitting proposed that capillary forces at the cathode caused by colloidal matter be considered as a cause of pitting. Romanoff (287) also studied pitting causes and found that emulsoid colloids caused the most difficulty in plating, but that electropositive colloids such as colloidal ferric hydroxide were probably those which cause pitting.

Macnaughton, Gardam and Hammond (332) investigated hard, small-grained deposits and found that they contained basic colloidal matter of a positive charge. Ballay (344), in investigating bright nickel produced by colloidal action, found that the brightness of the deposit

was connected with the existence of mineral or organic colloids in the cathode film. In ordinary baths it is possible to produce a bright deposit in a current density range just prior to the formation of burnt deposits. Ballay attributed this to colloidal hydroxide in the cathode film which is formed at the high pH of the cathode film caused by high current density. He also found that nickel plating solutions which contained colloids which pre-existed in the bath as organic colloids, gave more uniform results over a wide plating range, than those baths in which colloids were formed in the cathode zone. Kohlschutter (382) has also pointed out that the probably cause of "somatoid" elements of structure in electrodeposits is of colloidal origin. Macnaughton (386) followed up his earlier paper with his coworkers with one in which he stated that the hardness of nickel deposits was due to the difference in crystal structure caused by interference to normal growth caused by inclusion of basic nickel compounds, in colloidal state, which are produced at the cathode face as a result of local reduction of acidity. The quantity of colloidal matter thus formed, determined the properties of the deposit.

Another effect attributed to colloids was pointed out by Mayer (388) (422) who found that hydrophilic colloids were a cause of peeling. A theory by Glazunov and Drescher (414) attributed the effect of colloids to the reduction of the numerical value of the crystallization velocity, and pointed out that it was not dependent on an increase in viscosity. They felt that the effect of colloids is best explained by the assumption that complex cations are formed. Kochanoskii (450) found that nickel ions in solution were inactivated by the presence of

lyophilic colloids, and that the degree of activation was dependent directly on the degree with which the colloid was lyophilic.

In 1939 Macher (525) proposed a theory on the influence of colloids on the structure of electrodeposits which was quite unique. He proposed that colloids were adsorbed on the cathode during electrodeposition, and thereby formed a porous diaphragm with a"pin-cushion" structure. The discharge of the cations and the deposition of the metal he believed to occur in the intermicellar spaces in the diaphragm. The pores of the diaphragm were small and the member of crystal nuclei was large, and since the growth of the nuclei was hindered by the diffusion of the ions in the colloidal diaphragm, the deposit would be fine-grained.

Hendricks (627) has written an excellent article on the mechanism of bright electroplating. He believed that the deposition of bright nickel was a Liesegang phenomenon brought about by a periodic adsorption of a colloid, or a colloid mixture, thereby producing striated deposits of high reflectivity. He also ascribed the increase in cathode polarisation due to nickel brightneers to "a resistance through the colloid film caused by a porous colloid membrane. Even when the colloids are inorganic, the membrane will offer resistance since nickel ions from a complex with such colloids regardless of their charge, which travel by cataphoresis to the cathode." This idea of colloids migrating catapheretically to the cathode was earlier brought out by Hunt (314) who felt that if the proportion of metal ions to inert particles was low, due to the presence of colloids which had migrated catapheretically into the film or were codeposited there, the crystal growth would be altered. Glazunov (555) also proposed that

colloids which enveloped the cations and traveled to cathode by cataphoresis prevented the growth of crystals and increased the number of nuclei of crystallization on the cathode surface. He also proposed that the discharge of colloidal substances which combined chemically with metal resembled the discharge of complex ions, and since the deposition of the metal was a secondary process the deposit could not be made finer-grained by the presence of colloids.

Besides the articles mentioned, a number of other good articles are listed in the literature on the effects of colloids in electroplating solutions. These are by: Riedel (85), Millian (184), Frolich and Clark (200), Shikata and Hosaki (288), Glasstone (376), Cymboliste (410), Johnson (417), Meyer (423), (455), Stout (432), Vozdvizhenskii and Faizulin (433), Puri and Bhatia (533), Hamaker and Verwey (557), Puri and Jemeja (570), Puri and Seth (612), Snelling and Thews (613), Müller (631), Planner (635), and Bischer (662).

The Removal of Colloidal Matter from Nickel Plating Solutions: The removal of colloids from any solution is a problem since the particles are so small that they can not be removed by ordinary filtration. Some colloids can be precipitated by electrolytes, but it is assumed that colloids existing in nickel plating solutions would not be of this type since the solution is a strong electolyte. An article by Romanoff (287) appears to be the only one available on this subject. Romanoff suggested that "emulsoid" colloids could be removed by operating the bath at high current densities. He also recommended infusorial earths and similar materials as being valuable for filtering out colloids, of which colloidal iron hydroxide is a type, could sometimes be precipitated by

the action of strong oxidizing agents. Obviously, the colloids which are apt to cause the greatest difficulties in electroplating solutions are those which become positively charged and migrate to the cathod. High current density electrolysis is then one means of removing this type, although it is not apparently known to what degree of completeness the elimination may be carried out. It is possible that this process is too expensive in terms of the nickel also deposited out, to be practical.

A large percentage of the colloidal material found in electroplating baths is of organic composition. A good many colloids of this type can be successfully removed from nickel plating solutions by filtration through activated carbon. The elimination of metallic colloids, however, remains a difficult problem. There are, of course, methods for removing colloids from solutions such as dialysis or electrodialysis, but these methods would be impractical for application to electroplating solutions. Here again it is hoped that the experimental portion of this research will supply the answers to some of the unanswered questions on electroplating.

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