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POWER IN THE MANUFACTURE OF CHEMICALS AND METALS FROM BRINE

CONTENTS

Electrolysis releases from brines valuable chemical constituents essential to industry. Chlorine, metallic magnesium, and metallic sodium are produced in the United States today solely from brine chemicals by electrolytic methods. Caustic soda, hydrogen, bromine, sodium chlorate, and other chemicals are secured from brine by electrochemical methods, but are also produced by pyrochemical processes.

The passage of from 2,328 to 2,984 kilowatt-hours of direct current from the positive terminal through purified brine solution to the negative terminal of specific electrolytic cells produces simultaneously 1 ton caustic soda, approximately nine tenths ton chlorine, and approximately 8,100 cubic feet hydrogen. One ton metallic sodium requires over 13,000 kilowatt-hours energy when electrolyzed from fused caustic soda. Metallic magnesium production from dehydrated magnesium chloride is effected with approximately 16,000 kilowatt-hours of energy.

The electrolytic caustic soda and chlorine industry is firmly established; it consumed approximately 660,000,000 kilowatt-hours of energy in 1929. If all caustic soda sold in that year had been produced electrolytically, approximately 1,900,000,000 kilowatt-hours of electric current would have been consumed. Metallic sodium and other electrolytic sodium products are marketed in such small quantities that their production is not separately recorded.

The magnesium industry is in the initial stages of development in this country. Its increased production is primarily dependent upon the development of aeronautics. In 1929 approximately 11,000,000 kilowatt-hours of energy were consumed in its manufacture.

Because caustic soda and chlorine have served well-established and expanding markets for many years while other brine electrolytic chemicals and metals have had small markets, and also because some well brines contain from 96 to 98 percent sodium chloride and but small amounts of any one other chemical, the larger number of electrolytic plants have used only the sodium chloride content of brines. A beginning has been made, however, by one plant toward the complete utilization of the chemicals contained in complex brines. Natural brine containing 14 percent sodium chloride, 3 percent magnesium chloride, 0.15 percent bromine, and 9 percent calcium chloride is oxidized by electrolysis to separate out the bromine, which enters into dye, medicinal, and other chemical production. The sodium chloride crystallizes out of evaporated liquors and is separated into its component parts by electrolysis. The calcium chloride and magnesium chloride are separated, and the latter, after dehydration, is electrolyzed to metallic magnesium. A large number of valuable chemical products are manufactured from these basic brine constituents by combining them with such other basic chemicals as carbon, benzol, and sodium acetate. The total power generated at this chemical plant is 38,000 kilowatts.

Oceanic salts contain approximately 77.8 percent sodium chloride, 11 percent magnesium chloride, 5 percent magnesium sulphate, 2.5 percent potassium sulphate, and small amounts of magnesium bromide, calcium carbonate, and calcium sulphate. These salts have been evaporated for their sodium chloride content alone without recovery of other chemical constituents, along the California coast for many years. Only recently bromine and magnesium chloride are being produced from the accumulated bitterns on San Francisco Bay, and plans are under way to utilize the potash content of these bitterns. Unlike the recovery from the original brine, this reduction of bitterns is not an electrolytic process.

Handling of brine necessitates the evaporation of large quantities of water either before or after chemicals are produced electrolytically. While the market for dilute caustic soda is large, if the alkali is to be shipped any distance the cost of freight on dilute solutions must be balanced against the corresponding cost of more complete evaporation. Consequently the favored location of electrolytic alkali plants is near consuming markets, as well as near sources of raw materials. This condition has led to the erection of electrolytic caustic soda and chlorine units by consuming industries, such as pulp and paper mills, petroleum refineries, and detinning factories. It is seldom, however, that consuming industries can use to advantage in their own industry the three chemicals produced.

Chlorine and caustic soda find their principal consuming markets in the cellulose industries. These industries, as well as chemical industries, are affording expanding markets for both chemicals. Brine wells in New York State, abundant power, and a well-developed pulp and paper industry are responsible for the location of six electrolytic caustic soda and chloride plants in New York. Michigan electrolytic plants located above brine wells serve their own and Wisconsin pulp and paper industries. West Virginia electrolytic alkali plants serve principally organic chemical industries. Washington electrolytic plants have been established to serve the pulp and paper industry, while the California plant serves petroleum refineries and soap industries. The country's export market for alkalis lies in the Far East, a factor of importance in Pacific coast electrolytic alkali market development.

Sources and Chemical Composition of Raw Materials

1. Brine is the source of several chemical compounds which may be separated by electrolysis for use in industry. Obtained from sea water, from salt lakes, from salt desert and marsh lands, from salt springs, and from wells bored in rock-salt beds it contains varying amounts of sodium chloride in association with other chemicals.

2. *California.*—In oceanic salts, sodium chloride forms approximately 77.8 percent of the total salt content; magnesium chloride, 10.8 percent; magnesium sulphate, 4.7 percent; calcium sulphate, 3.6 percent; potassium sulphate, 2.5 percent; while less than 1 percent magnesium bromide and calcium carbonate are found. Solar evaporation is made possible by the long dry season of California along the shores of San Francisco Bay, Monterey Bay, San Pedro and San Diego Bays, and at other points along the coast where sea water is led into lagoons. Where salt has been evaporated for many years without the recovery of other chemicals, the bitterns contain accumulated amounts of magnesium chloride and magnesium sulphate as well as of bromine. Such bitterns are found in the San Francisco Bay region.

3. The brine of Owens Lake, Inyo County, Calif., in addition to sodium chloride, contains about 2 percent anhydrous boric acid and 2 percent potassium. Natural soda ash, sodium bicarbonate, and borax are produced. At Searles Lake, San Bernardino County, Calif., the brine averages 6 percent potash, 3 percent sodium tetraborate, 13 percent sulphate content, and contains iodine and bromine but little magnesium.[83] From this lake is produced much of the country's potash and borax.

4. *Utah.*—The Great Salt Lake of Utah contains 89 percent sodium chloride, 6.5 percent sulphate content, 2.5 percent magnesium, and 1.6 percent potash in its salts. Salt is produced at Saltair and at Garfield by solar evaporation.

5. What is claimed to be the most extensive rock-salt deposits in the world are located in the valley of the Sevier River near Salina, Utah. As the operating company has specialized in rock salt for livestock feeding, open-pit mining is employed. Salt springs occur in Juab County; and in Millard County and Tooele County crusts of salt appear in marsh and deserts where was once the ancient Lake Bonneville.

6. *Michigan.*—Michigan is the largest salt-producing State. Brine occurs in sandstone from 750 feet to 1,000 feet below the surface of the Saginaw Valley of Michigan. These brines are composed of approximately 83 percent sodium chloride, 3 to 7 percent magnesium, and from less than 1 percent to over 2 percent bromine. The calcine content is high in most beds, at times replacing the usual sodium content of 23 percent almost entirely. This brine is used largely for the manufacture of caustic soda, chlorine, soda ash, and other industrial chemicals.

7. The rock salt deposits which continue from the southeastern section of Ontario, Canada, under Lake Huron, under the St. Claire River, the Detroit River, and Lake St. Claire, yield a brine from bored wells 1,200 to 1,500 feet deep of approximately 94 percent sodium chloride and only small traces of other chemicals. Much the same composition is found in rock salt brines near Manistee.[84]

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[83] U.S. Geological Survey Bulletin 669, Salt Resources of the United States.

[84] Michigan Geological and Biological Survey Publication 35, Geological Series 29, Production and Value of Mineral Products in Michigan for 1923 and Prior Years.

8. *New York and Ohio.*—New York ranks second in salt production, Ohio, third. The rock salt beds which extend from Lake Erie eastward across New York State and for a short distance south into Ohio, contain from 96 to 98 percent sodium chloride and but small amounts of any one other chemical.[85] These are frequently mined by blasting. However, at Syracuse, N.Y., and Pomeroy, Ohio, magnesium-bearing bitterns are found. Such bitterns are found also at Hartford, W.Va.

9. *Kansas.*—Kansas ranks fourth in salt production. Rock-salt deposits occur in Ellsworth, Harper, Reno, and Rice Counties. This is marketed as rock salt or as evaporated salt made by running fresh water into bore-holes in rock salt beds and pumping the brine to the surface for heat evaporation.

10. *Other States.*—Louisiana also produces rock salt and evaporated salt. In Oklahoma some brine is obtained as a byproduct of oil wells. Texas produces evaporated salt. Small deposits of rock salt or brine are found in Arizona, southeastern Idaho, Nevada, New Mexico, Wyoming, Virginia, and West Virginia. Washington State produces sodium carbonate at Warden, Grant County.

11. Just how much of the brine and rock salt produced at 58 salt mining and evaporating plants in the United States enters into table salt, into ice cream freezing and packing, into cattle feed, meat packing, or hide salting, and how much is used by the electrolytic-alkali and ammonia-soda plants in the production of sodium and magnesium compounds is not ascertainable from brine sources. Less than 80,000 short tons of natural soda was produced in 1928, this coming entirely from Searles and Owens Lakes in California. Most of this natural soda was converted into sodium silicate or into caustic soda by the lime-soda process and was consumed by local industries. But there can be no doubt that the country's brine resources in the East, the Central, the Southern, the Rocky Mountain, and the Pacific coast sections are ample to meet expansion in sodium production as well as expansion of other markets in each section of the country.

12. Brines containing magnesium are more limited in occurrence. However, mineral deposits are available in Washington and California whenever it is commercially feasible to develop them for the magnesium metal market.

Electrolytic Products From Brine

13. Among the more important products resulting from brine electrolysis are caustic soda and chlorine, metallic sodium, and metallic magnesium. While other chemicals can be made in this way, it is more usual to produce them by purely chemical processes from caustic soda, metallic sodium, or soda ash.

14. As market demands are greatest for caustic soda and chlorine, major attention will be given these electrolytic chemicals.

CAUSTIC SODA AND CHLORINE

15. Since the industrial development of the caustic soda industry, its manufacture has been intertwined with the manufacture of a chlorine bleach.

[85] U.S. Geological Survey, Bulletin 669, Salt Resources of the United States.

HISTORICAL DEVELOPMENT IN ENGLAND AND FRANCE

16. In the eighteenth century the alkali necessary in the soap and glass industries was obtained by the lixiviation of ashes of seashore plants. The bleaching of textiles was achieved by exposure of textiles to the sun's rays. With the expansion of the several industries, these methods of obtaining chemical reactions were too slow and too uncertain to meet the growing demands. At the close of the century a Frenchman prepared a bleaching agent by dissolving gaseous chlorine in water, the chlorine being obtained by heating salt, manganese, and sulphuric acid in lead stills. At about the same time, a Frenchman named Nicolas LeBlanc prepared soda from common salt. By his process, salt was heated and treated with sulphuric acid, producing sodium sulphate or salt cake. This, in turn, was heated with coal and mixed with lime to produce sodium carbonate. Treatment with additional lime resulted in caustic soda.

17. The manufacture of both products, the alkali and the chlorine, was first developed commercially in England. However, Charles Tennant produced the first commercial bleaching powder by chlorinating hydrate of lime, thereby establishing the bleaching powder industry in England. The first successful alkali works, using the LeBlanc process, was erected in England in 1823. This process evolved hydrochloric acid gas which was wasted until Gossage, in 1836, condensed it and, by its reaction on manganese dioxide, produced chlorine. Gradually, this method of producing the bleaching gas superseded the earlier process upon which Tennant had established the bleaching powder industry in England.

18. The hydrochloric-acid gas produced in the LeBlanc process, however, exceeded the market requirements for chlorine, and much was allowed to escape into the air destroying all crops with which it came in contact. Or, when condensed, the acid was sent into streams killing the fish. Consequently, the LeBlanc process was in popular disfavor and many litigations resulted from its use. In 1863 and 1864 alkali acts were passed in England rendering compulsory the condensation of 95 percent of the hydrochloric-acid gas produced.

19. This situation made welcome an ammonia-soda process of manufacturing sodium carbonate invented by a Belgian chemical Engineer, Ernest Solvay. The Brunner-Mond Co., of England, which was producing sodium carbonate and chlorine products for bleaching powder by the LeBlanc process, adopted the new process whereby sodium carbonate could be produced in one operation and calcium chloride was the only waste product. The alkali was manufactured at a cost substantially lower than the cost of the older process although no chlorine for bleaching was obtained. Consequently, the cost of caustic soda and other sodium compounds was likewise reduced. So great was the success of the new process, as far as sodium compounds were concerned, that the LeBlanc producers were forced to unite and in 1890 formed the United Alkali Co., which is still in operation. Emphasis was placed upon chlorine bleaching powder production and salt cake rather than upon sodium carbonate and caustic soda by this organization. Recently, it has adopted electrolytic caustic soda and chlorine production methods.

DEVELOPMENT IN THE UNITED STATES

20. During these years of English dominance in bleaching powder and sodium compound production the United States imported most of her bleaching powder. As the demands for sodium compounds increased, ammonia-soda process plants were established on top of salt beds in New York, Ohio, and Michigan. These plants produced sodium carbonate as their base product and manufactured caustic soda and other compounds from it to meet trade demands. About 1890 Hamilton Castner succeeded in separating common salt into its two component parts by the passage of electric current through brine at Niagara Falls. The chlorine was given off as a gas while the sodium was collected in a mercury floor of the cell.

21. At about the same time the Elektron Co. of Germany had succeeded in producing electrolytic caustic soda and chlorine. Both the United States and Germany established protective tariffs to permit the infant electrolytic chlorine and caustic industries to grow in face of competition from England's LeBlanc bleaching powders.

22. The first American electrolytic plant was established at Rumford Falls, Maine, in 1893. In 1898 this plant was transferred to Berlin, N.H., to operated in conjunction with a paper pulp mill. In the same year the Castner Electrolytic Alkali Co. began making bleaching powder at Niagara Falls. The Dow Chemical Co. had started a 400-kilowatt plant to manufacture bleaching powder in 1897 at Midland, Mich. The Pennsylvania Salt Manufacturing Co. started operations at Wyandotte, Mich., in 1903. The Hooker Electrochemical Co. built an electrolytic plant at Niagara Falls in 1906.

23. Since that time other electrolytic plants have been established and ammonia-soda process plants have added electrolytic units to produce chlorine and caustic soda until there are 19 electrolytic plants having a capacity of 320,000 short tons of caustic soda and approximately 280,000 short tons of chlorine, with hydrogen as a byproduct. In addition, 17 pulp and paper mills have their own electrolytic alkali plants with an estimated capacity of 50,000 tons of caustic soda.

24. Prior to 1916 most of the chlorine produced in these plants was converted into bleaching powder by passing the chlorine over lime. It has been definitely established, however, that lime adds nothing to the bleaching qualities of chlorine and that, when in properly constructed containers, liquid chlorine lasts indefinitely while bleaching powder deteriorates rapidly. As methods of shipping chlorine safely have been perfected, the tendency of industries using large quantities of bleach has been to buy liquid chlorine in place of bleaching powder. Bleaching powder will continue to be used, of course, for smaller, package markets.

25. *Present production.*—The electrolytic separation of salt has become the prevailing method of producing chlorine, having almost succeeded in pushing the LeBlanc process material from the market, even in England. Only about a third of the caustic soda manufactured, however, is made electrolytically. The demands for soda ash (sodium carbonate) so far exceed the market for caustic soda that five ammonia-soda companies have been able to control caustic soda prices. These plants produce the caustic soda from sodium carbonate by treatment with lime.

26. *Caustic soda.*—The increased demand for caustic soda shown on table I of approximately 128 percent since 1919, or from 333,000 tons to 758,800 short tons has been met by an increase in electrolytic production proportionate only to the increase in lime-caustic soda production.

27. Nor does the production of 758,800 short tons represent the total market demand for caustic soda. In soap and paper industries requiring large amounts of caustic soda in dilute solution large producers buy soda ash and causticize it themselves. As a consequence, part of the soda-ash market is really a caustic-soda market. Nor is the production of the 17 pulp and paper mills manufacturing electrolytic caustic soda and chlorine for their own requirements included, although it is estimated this would add from 30,000 tons to 50,000 tons to the electrolytic caustic-soda production.

TABLE I.—*Production of caustic soda (sodium hydroxide) in the United States* [1]

Year	Total production (short tons)	Percent increase or decrease over 1919	Electrolytic process [2]		Lime caustic soda	
			Short tons	Percent of total	Short tons	Percent of total
1919	333,361		75,547	31.7	163,044	68.3
1921	238,591	−28.4	122,424	28.0	314,195	72.0
1923	436,619	+31.0	141,478	28.5	355,783	71.5
1925	497,261	+49.2	186,182	32.5	387,235	67.5
1927	573,417	+72.0	233,815	30.8	524,985	69.2
1929	758,800	+127.6				

[1] U.S. Bureau of the Census: Chemicals.
[2] Does not include tonnage manufactured and consumed at pulp and paper mills estimated to be approximately 30,000 tons in 1927 and 1929.

28. *Chlorine.*—The failure of electrolytic caustic soda to secure a more important place in the alkali market has been due largely to its dependence on the market for chlorine. For every ton of caustic soda produced approximately nine tenths of a ton of chlorine is obtained that must find a profitable market. Fortunately, for the continued success of the electrolytic industry, the demand for chlorine is increasing. It is difficult, however, to determine the exact amounts of chlorine entering industry from census figures for firms producing chlorine convert some into bleaching powder, or into hydrochloric acid, and several manufacture a number of other chlorine compounds.

29. On the basis of electrolytic caustic soda produced, the production of chloride was probably approximately 210,000 short tons. The chlorine marketed as compressed and liquefied gas totaled 120,648 tons, an increase of 165 percent over 1919 production and a 45 percent increase over 1925. Bleaching powder manufacture, including sodium hypochlorite, decreased 0.2 percent since 1919, or a little over 119,886 tons were manufactured in 1929.

TABLE II.—*Production of chlorine and bleaching powder in the United States* [1]

Year	Chlorine		Bleaching powder and sodium hypochlorite	
	Total production (short tons)	Percent increase or decrease over 1919	Total production (short tons)	Percent increase or decrease over 1919
1919	45,571		126,425	
1921	37,985	−16.6	86,602	−31.5
1923	62,681	+37.5	157,914	+24.9
1925	83,163	+82.5	130,830	+3.5
1927	117,489	+157.8	133,642	+5.7
1929	120,648	+164.7	119,886	−5.2

[1] U.S. Bureau of the Census. Compressed Gases, and Chemicals.

30. *Producing plants and their locations.*—Plants at which electrolytic caustic soda and chlorine are produced are located in the following towns and cities:

California:	Pittsburg, Great Western Electrochemical Co.
Illinois:	East St. Louis, Monsanto Chemical Works.
Michigan:	Midland, Dow Chemical Co.
	Wyandotte, Pennsylvania Salt Manufacturing Co.
New Jersey:	Deepwater Point, E. I. duPont de Nemours & Co.
New York:	Niagara Falls, Hooker Electrochemical Co., Isco Chemical Co., Mathieson Alkali Works, Niagara Alkali Co., Niagara Smelting Corporation.
	Syracuse, Solvay Process Co.
Ohio:	Painesville, Diamond Alkali Co.
Pennsylvania:	Pittsburgh, Vulcan Detinning Co.
Texas:	Port Arthur, Gulf Refining Co.
Washington:	Tacoma, Hooker Electrochemical Co., Pennsylvania Salt Manufacturing Co.
West Virginia:	Belle, Belle Alkali Co.
	Nitro, Monsanto Chemical Works.
	South Charleston, Westvaco Chlorine Products Co.

31. Pulp and paper mills operating their own electrolytic alkali plants are:

Delaware:	Wilmington, Jessup and Moore Paper Co.
Maine:	Cumberland, S. D. Warren & Co.
	Great Works, Penobscot Chemical Fibre Co.
	Rumford, Oxford Paper Co.
	South Brewer, Eastern Manufacturing Co.
Maryland:	Elkton, Jessup & Moore Paper Co.
	Luke, West Virginia Pulp & Paper Co.
New Hampshire:	Berlin, The Brown Co.
New York:	Mechanicsville, West Virginia Pulp & Paper Co.
North Carolina:	Canton, Champion Fibre Co.
Ohio:	Carrollton, Miami Paper Co.
Pennsylvania:	Johnsonburg, New York & Pennsylvania Paper Co.
	Philadelphia, Dill & Collins.
	Roaring Springs, D. M. Bare Paper Co.
	Tyrone, West Virginia Pulp & Paper Co.
Virginia:	Covington, West Virginia Pulp & Paper Co.
Wisconsin:	Kimberly, Kimberly-Clark Co.

32. Ammonia-soda alkali plants producing lime-caustic soda are:

Kansas:	Hutchinson, Solvay Process Co. (not operating).
Michigan:	Detroit, Solvay Process Co.
	Wyandotte, Michigan Alkali Co.
New York:	Syracuse, Solvay Process Co.
Ohio:	Barberton, Columbia Chemical Co.
	Painesville, Diamond Alkali Co.
Virginia:	Saltville, Mathieson Alkali Co.

Electrolytic Caustic Soda and Chlorine Process

32. A pure solution of common salt (sodium chloride) forms the electrolyte for this process. Consequently, the first step is the purification of the raw material. Methods will vary with the type salt used. When magnesium and lime are present, the magnesium is removed by adding waste salt containing caustic soda; and lime is removed with soda ash. The purified brine is filtered and a little hydrochloric acid added. As the brine solution enters the electrolytic tank it has a salt content of approximately 26 percent.

33. The passage of a direct-electric current from a positive terminal or anode through a sodium-chloride solution to the negative terminal or cathode decomposes the salt into its component parts, sodium and chlorine. When chlorine and caustic soda (sodium hydroxide) are the desired products, electrolytic cells are used that prevent secondary reactions of these chemicals after their dissociation. Methods of effecting the separation of these chemicals vary in the different types of cells. The cells in most frequent use in this country are of the diaphragm and of the moving mercury cathode types.

34. *Diaphragm cells.*—In all diaphragm cells porous diaphragms separate the anode compartment from the cathode compartment. Liquor may be on both sides or on only one side of the diaphragms. The brine enters the anode compartment and percolates through diaphragms to the cathode sections. The liberated chlorine gas escapes at the anode into collection mains, while the sodium is deposited on the cathode, where it reacts with water in the submerged type of cell to form sodium hydroxide (or caustic soda) and hydrogen. The hydrogen is carried off and the caustic soda solution drops into troughs or pockets. In some tanks the cathode is not immersed but is only wetted by the percolations through the diaphragm and the alkali is washed away with steam.

35. In another type of cell, the cathode compartment is filled with high-boiling mineral oil. The brine percolates slowly through the diaphragm into the oil. The caustic soda is thrown off and sinks, while hydrogen bubbles rise to the oil surface and are carried off. In a third type of diaphragm cell a cylindrical form is used, and only one side of the diaphragm is submerged. The anode is made up of graphite strips fastened to the concrete asbestos anode cover. The cathode is a perforated iron cylinder around which the diaphragm of asbestos is wrapped.

36. Diaphragms in all types are usually of asbestos, although paper pulp has been used. Anodes are of electric-furnace graphite, and cathodes are made of perforated iron. Graphite anodes usually last a year.

37. The current density determines, to a large extent, the cell voltage required and, consequently, the production per unit of electricity. While a high current density will give greater output per cell, a low current density will require less energy per unit produced. The current density employed is determined by the particular cell in use and the cost of power. The cell having mineral oil in the cathode compartment takes highest current densities of the prevailing diaphragm cells. At 3,000 amperes and 4.05 volts it will produce 225 pounds caustic soda in 24 hours at a cost of 296 kilowatt-hours, while a 4,000-ampere cell at 4.65 volts would produce the same amount in

18 hours at a cost of 336 kilowatt-hours, or 1 ton of caustic soda would require 2,630 kilowatt-hours at the lower density and 2,984 kilowatt-hours at the higher density.

38. The cells having unsubmerged diaphragms usually require from 1,000 amperes to 1,500 amperes and a voltage of 3.6 to 3.9. Current efficiency in new cells is from 92 to 95 percent. The energy consumption per ton caustic soda would be from 2,340 to 2,500 kilowatt-hours. About nine tenths ton of chlorine and 8,100 cubic feet of hydrogen are produced at the same time.

39. A plant using the circular diaphragm cells having 95 percent current efficiency, with an average voltage of 3.5 and an average amperage of 948.1 per cell produced 0.859 pound of caustic soda and 0.793 pound of chlorine per kilowatt-hour, or the production of 1 ton of caustic soda and 1,846 pounds of chloride required 2,328 kilowatt-hours. Per ton of finished product, exclusive of hydrogen, 1,210 kilowatt-hours are consumed.

40. *Mercury cells.*—In the mercury cathode cell two slate partitions divide the cell into three sections which are sealed from each other by mercury covering the tank bottom. The central section contains an iron cathode, made up of strips, and is filled with water. The outer chambers have graphite anodes and are filled with brine. The chlorine is carried off as in other cells, but the sodium combines with the mercury. The mercury is circulated by an Archimedean screw or by tilting the tank slowly. As the sodium and amalgam pass to the center chamber, they act as the anode and water combines with the sodium to form caustic soda and hydrogen.

41. These cells require on an average 4,000 amperes at 4.4 volts. A 40-percent caustic solution is obtained which is a higher concentration than that gotten in diaphragm cells. The caustic soda is also very pure. But power requirements are 2,900 kilowatt-hours per ton of caustic soda and per 1,760 pounds of chlorine, or higher than in prevailing diaphragm cells. Mercury costs are also considerable items in this process.

42. The I. G. Farbenindustrie of Germany has patented a process whereby a 60-percent concentration of caustic soda is obtained through the agitation of the sodium amalgam with nitrobenzene and water.

43. *Evaporation of caustic soda solution.*—The solution from all diaphragm cells contains more salt than caustic soda. Consequently, evaporation must not only concentrate the caustic soda but free it of salt content. This is accomplished in double-effect, single, or triple-effect evaporators, operating under vacuum and heated by steam. In new plants the process is continuous and circulation is forced; in older plants it is a batch cycle requiring 5 or 6 hours to produce 12 tons of 50 percent caustic. The weak liquor is gradually concentrated to 50 percent caustic soda solution. This is pumped to crystallizers where cooling by a countercurrent flow of water in the jackets causes the salt to crystallize out. The salt is washed thoroughly and sent to settling tanks, ready to be used again in the purification of raw brine.

44. Much caustic soda is now marketed in tank cars as a 50 percent solution. The remainder is further evaporated in open fusion pots to solid and flake caustic.

45. During a 24-hour test run of a modern evaporating plant, 53 tons of 100 percent custic soda were evaporated from 1,026,000 pounds

of water using 746,000 pounds condensed steam at 10-pound gage pressure. The condensers required on an average of 2,200 gallons of cooling water per minute. Power consumption of the pumps which forced liquor circulation totaled 90 kilowatts per hour.[86] Or approximately 40 kilowatts hour of energy must be added to each ton of caustic soda produced for evaporator-pump operation. About 120 additional kilowatt-hours are consumed by the evaporating plant in lighting and in miscellaneous uses.

46. Although shipping 50 percent caustic soda to consumption plants lessens evaporation costs materially, efforts are being made to isolate caustic soda from the salt solution and to evaporate the weak caustic soda liquor by speedier and less expensive methods. The department of chemical engineering of the University of Michigan has dehydrated caustic soda in a forced circulation evaporation system by use of diphenyl vapor as a heating medium, thereby eliminating the pot-finishing process. A patent by D. A. Pritchard, of the United Alkali Co., of England, adds salts of sulphuric acid to precipitate the sodium chloride.

47. *Liquid chlorine.*—Chlorine gas withdrawn from the top of the electrolytic cells into stoneware collection mains is carried through cooling coils to drying towers. Here it passes against 66° Baumé sulphuric acid to remove moisture. The dry chlorine gas is delivered to liquefaction towers. It is entrained with a falling column of strong acid. Falling through a distance of approximately 90 feet into a compression chamber, gas pressure is increased and as gas rises through cooling coils it liquefies. The liquid chlorine is run into steel vessels and from there into tank cars for market, or into storage tanks.

48. *Bleaching powder.*—Some chlorine is still converted into bleaching powder. Hydrated lime is placed in coil-cooled concrete chambers to a depth of several inches. The chambers are sealed and chlorine gas and air are passed over the lime countercurrently. The chloride is absorbed by the lime to make chloride of lime or bleaching powder.

49. *Hydrochloric acid.*—An acid-proof refractory furnace is used as the reaction chamber when chlorine is made into hydrochloric acid. Chlorine and steam enter in the furnace bottom while coke is charged in at the top. The mixture passes through dust chambers, is cooled, and gases are absorbed in a tower system. The acid may be made directly from sodium chloride by the action of strong sulphuric acid in mechanical furnaces, producing sodium sulphate as a byproduct.

50. *Synthetic ammonia.*—Byproduct hydrogen (amounting to approximately 9,000 cubic feet per ton of chlorine produced is collected and metered. It is mixed with air in proportion of 75 to 25 and oxygen is burnt out. The residual gases are cooled and scrubbed to remove impurities. They are compressed at pressure of 4,000 pounds. At this pressure the nitrogen-hydrogen mixture passes over a soda-lime tower for further purification. The gases are then ready to be catalytically combined by a modified Haber process. (For details of Haber process see pp. 142 and 143, of Power in Fertilizer Material Production.) For this method of ammonia production from 85,000 to 100,000 cubic feet of hydrogen are required, 75,000 cubic feet to

[86] Lee, James A. Westvaco Sets New Record in Evaporating Electrolytic Caustic Soda. Chemical and Metallurgical Engineering, July 1930.

be used as hydrogen in the catalytic chambers and 10,000 to 25,000 cubic feet to secure nitrogen from air.[87]

Ammonia-Soda Process

51. The basic raw materials for the ammonia-soda process are salt, limestone, coke, and coal. Ammonia may be produced at the plant from water-gas hydrogen and producer gas nitrogen from the coke, or from coke-oven hydrogen and liquefied nitrogen (see pp. 144 and 145 of Power in Fertilizer Material Production), or it may be purchased. Carbon dioxide is obtained through the burning of limestone to produce lime and also from the recovery of ammonia from the mother liquors. One ton of sodium carbonate requires 253 pounds of coke, 2,645.5 pounds limestone, 1,102 pounds coal, and 5 pounds ammonia.[88] Large producers who own their own limestone quarries and are on waterways have been able to get limestone delivered for less than $0.70 per ton stone. Some plants also have their own coke ovens.[89]

52. Strong brine, after purification, is saturated with ammonia gas in saturator towers. This ammoniacal brine is cooled and then passes to carbonating towers. In these carbon dioxide is pumped in at the base and ascends the tower as ammoniacal brine descends. In the lower sections of the tower are cooling tubes. Usually the mixture passes to another tower, in which ammonium chloride (NH_4Cl) is formed and soda bicarbonate precipitated. The soda bicarbonate is filtered in vacuum filters and is calcined to sodium carbonate ($NaCO_3$). If bicarbonate of soda is desired, all ammonia is driven off the sodium is recarbonated, and is dried in a hot air current.

53. The ammonium chloride may be converted into calcium chloride with lime. Or, if ammonia is desired for continual use in the manufacture of sodium carbonate, it is recovered in distillery towers. Steam entering at the base dries off the carbon-dioxide gas. Milk of lime entering halfway up the tower frees the ammonia from the chlorine.

54. *Lime caustic soda.*—The tank liquor containing about 660 parts of sodium carbonate in 2,000 parts of water is heated to 90° C. Three hundred parts of quicklime are added gradually as the solution is stirred. This is boiled for about an hour, when the liquor filtered off from the calcium carbonate contains about 14.5 percent caustic soda and over 7 percent sodium carbonate. By a gradual cooling in a tank having cold brine circulating coils or other cooling systems, the sodium carbonate crystallizes out. At a temperature of minus 15° C. only about 0.8 percent sodium carbonate remains in the solution while the caustic soda forms 18 percent of the solution. The sodium carbonate crystals are recovered for reuse in caustic soda production. The caustic liquor is evaporated by waste heat and in soda pots or through a multiple-effect vacuum evaporating system as in electrolytic plants. One and thirty-five hundredths (1.35) tons of soda ash is required for 1 ton caustic soda.

55. *Causticized caustic soda.*—Crude sodium is calcined to a 40 percent sodium oxide (Na_2O). This is saturated in a rotary drum

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[87] Manning, Paul D. Pacific Coast Industry Diversifies Products, Chemical and Metallurgical Engineering, June 1930, specified 100,000 cubic feet.

[88] Partington, J. R. The Alkali Industry, p. 127.

[89] Hughes, L. C. Modern Ammonia-Soda Plants. Chemical and Metallurgical Engineering, April 1930, p. 232.

and mixed with milk of lime. After passing through thickeners it goes to causticizing tanks where it is kept in agitation. In 1 hour a 90 to 91 percent caustic is secured. This passes through several thickeners and filters and finally goes to brine kilns. One ton of lime is used per ton caustic produced. Calcium carbonate is a byproduct.

56. *Other chemicals produced at electrolytic plants.*—As caustic soda or chlorine are raw materials for a number of other chemicals, as some brines contain chemicals other than sodium chloride, it is not surprising to find that electrolytic producers for the general market manufacture such sodium and chlorine compounds, or such other brine chemicals, as their market demands.

57. The Great Western Electro-Chemical Co. of California, in addition to 40 tons electrolytic caustic soda, 35 tons chlorine, 27 tons bleaching powder, 5 tons synthetic anhydrous ammonia, and 1.5 tons aqua ammonia, produces, as the market requires, hydrochloric acid, sodium hypochlorite, sodium xanthate, potassium xanthate and alkyl xanthate, ferric chloride, zinc chloride, and diammonium phosphate fertilizer.

58. While the new Tacoma plant of the Hooker Electrochemical Co. is at present confinding its manufacture to two basic chemicals, at the parent plant in Niagara Falls, the following additional chemicals are produced: Hydrochloric acid, monochlorbenzene, paradichlorbenzene, benzoate of soda, benzoic acid, benzoyl chloride, benzyl alcohol, benzyl chloride, antimony trichloride, ferric chloride, sulphur monochloride, sulphur dichloride, sulfuryl chloride, and table salt.

59. The Dow Chemical Co. produces from complex Michigan brines more than 45 chemicals. The bromine and the magnesium content of these brines is manufactured into bromides and ethylene bibromide, metal magnesium, and magnesium chloride, and arsenate, while the caustic soda and chlorine are treated with other basic materials to produce such chemicals as chlorbenzol, phenol, salicylic acid, and aspirin; carbon tetrachloride, chloroform, synthetic indigoes, and synthetic perfumes; ferric chloride, chloracetic acid, and acetic andydride used in cellulose acetates. Salt and metallic sodium are also among the many products.

60. The production of these three companies is illustrative of the interlocking chain of chemicals that may be manufactured in electrolytic caustic and chlorine plants in the several sections of the country.

61. *Cost of production.*—The multiplicity of chemicals manufactured at electrolytic caustic and chlorine plants permits a part of the cost of production of the basic chemicals to be borne by a number of materials having much greater market value than the base chemicals. The diversification of products in the several factories makes it impossible to determine the maximum cost of production of caustic and chloride that any individual plant may reach and yet compete successfully with other plants in its specific markets. The price of caustic soda is still determined by ammonia-soda plants in which caustic soda is almost a by-product.

62. Capital costs of electrolytic production are relatively high, especially when firms own chlorine tank cars. Engineers from firms operating such cars claim they represent an investment of almost $50 per ton per year. Where hauls are short and turnover of car capacity frequent, this unit cost would be materially reduced.[90]

[90] Transactions of American Electrochemical Society, 1926, p. 57.

63. Costs of raw materials are low unless salt carries heavy freight charges. Standard ocean freight rates run from $2.50 to $3 per ton, although coastwise steamers, wishing ballast cargo, accept a much lower rate.

64. With a total plant power consumption of from 1,350 to 1,704 kilowatt-hours per ton of caustic and per ton of chlorine produced, at a $0.003 rate, power costs would total but $4 to $5 per ton. Labor costs represent a larger item in total electrolytic plant costs than does power at this rate; however, this unit labor cost decreases with the size of the plant and varies with the character of additional chemicals manufactured. Renewals of equipment and plant maintenance costs are relatively high.

65. The location of any particular plant in relation to its sources of raw material and to its finished product markets are, therefore, more vitally important to its competitive success, at this time, than other items. Caustic soda, 76 percent solid, sold for from $58 to $60 a ton last year. In January 1931 the price had dropped to $45. Liquid chlorine brought from $12 to $14.75. Any hydrogen that could be marketed as such decreased the total cost by the price it brought.

66. *Caustic-soda consumption.*—The heavy increase in consumption of caustic soda between 1927 and 1929 was due primarily to the growth of the rayon industry. Increased demands came also from petroleum refineries, rubber reclaiming, pulp and paper, soap, and chemical industries. Table III, page 295, shows the estimated consumption by industries of caustic soda and soda ash in 1929 and 1930. The lessened consumption in 1930 is due to general industrial depression. Consequently, 1929 figures are regarded as better indices of alkali consumption markets.

67. The largest markets for caustic soda are offered, at present, by the rayon, the chemical, the petroleum, and the soap industries, each of which purchases 15 percent or more of the total amount marketed.

68. *Caustic soda in pulp production.*—While the pulp and paper industry bought from but 6 to 7 percent of the marketed caustic soda, a number of pulp mills produce caustic soda for their own needs either electrolytically or by causticizing soda ash. Caustic soda is essential to pulp manufactured by the soda process, as this process depends upon caustic soda to dissolve certain constituents of the wood as well as to hydrochlorize other constituents. Several of the newer methods of producing new types of paper also require caustic soda. It is probable, therefore, that the pulp and paper industry actually consumed about as much caustic soda as the other principal consuming industries.

69. *Caustic soda in rayon industry.*—Caustic soda is used in the Viscose process of manufacturing rayon to form cellulose xanthate which is decomposed. As the largest amount of rayon is now produced by this process and as it is believed this production will increase, unquestionably rayon will represent an increasing market for caustic soda.

70. *Caustic soda in soap manufacturing.*—All soaps require an alkali. This may be lye, made from caustic soda, sodium silicate made from soda ash, soda ash, or caustic soda, depending upon the fatty acid used and upon the kind of soap desired.

71. *Caustic soda in rubber reclaiming.*—Some 40,000 tons of caustic soda are used in rubber reclaiming. Production of reclaimed rubber

in the United States increased from 208,516 long tons in 1928 to 219,057 tons in 1929, while our exports increased from 9,577 to 12,721 tons. We export to 26 countries. Reclaimed rubber constitutes almost half of our total rubber consumption.[91] In this connection it is interesting to note that the Roessler & Hasslacher Chemical Co., which manufactures electrolytic caustic soda at a subsidiary at Niagara Falls reclaims rubber at El Morete, Calif.

72. *Shifting markets.*—Recently, a chemical market for caustic soda was successfully claimed by lime, a much cheaper alkali than caustic soda. This incident drew attention to the fact that perfection of processes in every industry, together with greater scientific data concerning the specific characteristics of each alkali in industrial application, may cause other adjustments in well established markets for specific alkalis. The increased production of ammonia as a result of the development, on a large scale, of direct synthetic ammonia plants in this country has led to a vigorous effort on the part of large ammonia manufacturers to find markets for ammonia as an alkali. Research is now underway to determine the possibility of substituting ammonia for other alkalis in the paper industry.

73. The electrolytic caustic soda manufacturer may be said to be protected from any such competition because hydrogen, the more costly constituent of ammonia, is a byproduct of electrolytic caustic soda production and is now being manufactured into synthetic ammonia at several electrolytic caustic soda plants.

TABLE III.—*Estimated consumption of caustic soda and soda ash in 1929 and in 1930* [1]

Industry	Caustic soda (sodium hydroxide)				Soda ash (sodium carbonate)			
	1929		1930		1929		1930	
	Short tons	Percent	Short tons	Percent	Short tons	Percent	Short tons	Percent
Total	750,000	100.0	640,000	100.0	1,668,000	100.0	1,475,000	100.0
Chemicals	135,000	18.0	100,000	15.6	335,000	20.0	290,000	19.7
Petroleum refining	121,000	16.1	105,000	16.4	26,000	1.6	22,000	1.5
Rayon	115,000	15.3	110,000	17.2				
Soap	108,000	14.4	100,000	15.6	213,000	12.8	200,000	13.6
Exports	60,000	8.0	63,000	9.8	40,000	2.4	36,000	2.4
Pulp and paper	[2] 45,000	6.0	[2] 42,000	6.6	110,000	6.6	100,000	6.8
Textiles	42,000	5.6	30,000	4.7	40,000	2.4	30,000	2.0
Rubber reclaiming	40,000	5.3	20,000	3.1				
Lye	25,000	3.3	22,000	3.4				
Vegetable oils	11,000	1.5	10,000	1.6				
Glass works					672,000	40.3	590,000	40.0
Cleansers and modified					125,000	7.5	112,000	7.6
Water softeners					60,000	3.6	55,000	3.7
Miscellaneous	48,000	6.4	38,000	5.9	47,000	2.8	40,000	2.7

[1] Compiled from estimates made by Chemical and Metallurgical Engineering, January 1931.
[2] Does not include caustic soda manufactured by pulp and paper mills.

CONSUMPTION OF CHLORINE

74. *Well-established chlorine markets.*—Although exact figures concerning chlorine consumption are not available, it is estimated that almost two thirds of all chlorine produced enters the pulp-and-paper

[91] U.S. Bureau of Foreign and Domestic Commerce, Rubber. June 1930.

industry. Whether in liquid or gaseous form, as a hypochlorite, or in bleaching powder, some form of chlorine is used commercially to remove undesirable impurities which are left by any method of isolating fibrous cellulose for paper.

75. Over 20 percent of chlorine produced is believed to be used in textile bleaching. In Europe, chlorine is used to degrease wool, but in the United States such grease is not recovered at all or recovered by solvent naphtha. In the bleaching of cotton, silk, and wool, chlorine may face competition with hydrogen peroxide.

76. With the increasing density of population, the requirements for chlorine or chlorine compounds in drinking water, as a purifier for bathing beaches and swimming pools, and to treat sewage and garbage odors are growing. The addition of ammonia to the chlorine used in drinking-water purification, to eliminate chlorine taste, may reduce the unit consumption of chlorine, but the increased need for adequate sanitation will unquestionably lead to an increased use of chloride compounds in populated areas. D. A. Pritchard of the Canadian Salt Co., Ltd., estimates 10 percent of all chlorine produced was required for sanitation measures in 1926.

77. *New chlorine markets.*—Chlorine, as sodium or calcium hypochlorites, has been used in the petroleum industry to remove objectionable odors from gasoline and kerosene. The Gulf Refining Co. at Port Arthur, Tex., has just perfected a method of producing a cheap supply of aluminum chloride for oil refining. This company has its own electrolytic chlorine plant. Bauxite is reduced by carbon and chlorinated simultaneously. Anhydrous aluminum chloride can be produced at the rate of 75,000 pounds per day at a cost of less than $0.05 per pound.

78. Another plant that has installed an electrolytic chlorine plant for its own use is the Vulcan Detinning Co. of Pittsburgh. Chlorine is used to detin tin-plate scrap, as chlorine gas will combine with tin under heat to form anhydrous tin tetrachloride. This is sent to the silk industry for weighting while steel scrap is available for steel furnaces.

79. The largest recent increased demand for chlorine has come from the organic chemical industry. Although employment of chlorine in organic chemicals—such as chloroform, carbon tetrachlorate, and chlorobenzene—has been carried on for many years, the more recent growth of the synthetic organic chemical industry in this country is responsible for the new demands.

80. Chlorine will react with many organic compounds. In the aliphatic series developed upon the segregation of component gases in natural gas, chlorine or its compounds may be used to form additional products, such as result from its combination with acetylene, to form substitution or oxidation products, or it may be used in combination with other elements. For example: Ethylene, a hydrocarbon gas, may be combined with hypochlorous acid to form ethylene chlorhydrin. Or it may be converted into ethylene chloride. Or, by the direct chlorination of methane, methlychloride, isopropyl, secondary butyl, secondary amyl, and secondary hexyl alcohol are obtainable. The greater demand for domestic electric refrigerators has led to a slow but increasing use of methyl chloride as the refrigerant in small-capacity refrigerators. In this field it has come in direct com-

petition with sulphur dioxide which was first used in household refrigerants.

81. Other organic chemicals in which chlorine is employed are monochloracetic, chloracetyl chloride, propylene chlorhydrin, ethylene dichloride, and dichlorodiethyl sulphite. Some chlorine organic chemicals have found ready markets; for others, markets must still be found.

82. *Chemical warfare.*—Consideration of chlorine markets would not be complete without recognition of the part chlorine plays in chemical warfare. According to Maj. Gen. Amos Fries, formerly head of our Chemical Warfare Service, chlorine is the basic ingredient of almost every one of modern chemical warfare agents. In phosgene, mustard gas, chloropicrin, tear gases, toxic smokes, and obscuring smokes, chlorine either appears in the product used or is essential to its manufacture.

83. The outlook for chlorine seems excellent, for with but minor competition in its well-established markets, its possibilities for increased use with the growth of the synthetic organic chemical industry are great.

84. *Location of consuming markets.*—The many products into which caustic soda and chlorine enter make it difficult to ascertain the amount of each chemical consumed in particular locations. By a process of weighting each State in accordance with its importance in the industries which are the heaviest consumers of each chemical, the relative size of the consuming markets have been ascertained.

85. Beyond a doubt, New York and Pennsylvania offer the largest existing markets for chlorine and caustic soda. Maine, because of its soda-pulp manufacture for which it requires caustic soda and because of its large consumption of chloride in all types of pulp production; New Jersey and Ohio, because of their chemical, soap, rubber, and miscellaneous industries; all three are heavy consumers of the heavy chemical and the gas.

86. The Pacific coast and such southern States as Virginia, West, Virginia, and Tennessee give promise of rapidly developing heavy markets for these chemicals. The South, because of its expansion as a rayon and chemical center must needs increase its requirements for caustic and chlorine. The diversification of wood-pulp production in the Pacific Northwest, which will not only increase the types of pulp made for paper but develop pulp for rayon and other cellulose-solution products, will bring with it an increased need of chlorine and caustic soda. The developments in gas cracking which have begun in California may lead to an organic chemical industry on this coast. The normal expansion of rubber reclaiming, of soap manufacture, of petroleum refining, in orchard spraying, and in the cyanide and flotation process for mining will call for chemicals of which one or the other of these electrolytic chemicals form the base.

87. *The export market.*—About 8 percent of the total caustic soda produced in this country is exported. Over a third of these exports is to Japan. Japan has a number of her own electrolytic caustic soda and chlorine plants but must import salt from Manchuria, Tsington, Spain, or Egypt at an average cost of $10 per ton.[92] While Japan

[92] Ochi, Shuichiro. Chlorine in Japan, in Transactions of the American Electrochemical Society, vol. XLIX, 1926.

has consumed caustic and bleach, chiefly in the pulp and textile industries in the past, the present development of viscose rayon manufacture will, unquestionably, increase her caustic requirements. Mexico, Cuba, Canada, the Philippines, and South American countries also buy caustic soda from the United States.

Chlorine and bleaching powder are shipped in small quantities to Canada and to South and Central America.

88. The United States has no European market for caustic or chlorine. Its export market lies principally in the Far East and in South and Central America. Here it comes in competition with Great Britain, which supplies about three fourths of the present market.

TABLE IV.—*Exports of sodium and chlorine products in 1929, by country of destination* [1]

Country	Caustic soda		Modified soda products		Chlorine		Bleaching powder	
	Short tons	Value	Short tons	Value	Short tons	Value	Short tons	Value
Total	60, 538	$3, 516, 053	7, 497	$424, 541	3, 584	$256, 667	2, 512	$127, 767
Argentina	6, 331	436, 723	620	31, 208	-------	40	650	17, 780
Brazil	3, 187	211, 886	855	54, 370	6	4, 220	63	9, 607
British India	852	52, 942	36	3, 423	-------	72	-------	-------
Canada	3, 825	270, 542	1, 415	74, 617	3, 339	196, 943	1, 353	61, 876
Chile	357	18, 514	252	12, 776	32	10, 713	1	87
China	2, 400	134, 018	507	27, 360	45	7, 608	1	67
Colombia	677	40, 525	198	11, 921	36	9, 452	65	1, 971
Cuba	4, 492	273, 117	410	24, 802	26	4, 012	242	25, 753
Japan	22, 657	1, 162, 048	808	43, 434	-------	40	-------	-------
Java and Madura	2, 191	113, 938	306	15, 850	-------	-------	-------	-------
Mexico	7, 450	446, 005	944	51, 136	26	7, 349	32	1, 736
Netherland West Indies	828	43, 846	123	6, 749	-------	71	1	299
Philippine Islands	2, 730	143, 076	507	25, 044	39	6, 077	41	2, 401
Other countries	2, 561	168, 873	516	41, 851	35	10, 070	63	6, 190

[1] Compiled from complete reports on exports and imports of the United States in 1929, which appear in Foreign Commerce and Navigation of the United States, U.S. Department of Commerce.

OTHER ELECTROLYTIC SODIUM PRODUCTS

89. *Metallic sodium.*—The world production of metallic sodium is estimated to be approximately 27,500 short tons by Dr. Hess, manager of the Dr. Alexander Walker Co., of Munich, Bavaria. Of this amount, Germany produces 11,000 short tons. American firms manufacturing it market but little, as it is used by them chiefly to produce sodium peroxide, sodium cyanide, or a synthetic indigo. Its ready oxidizability makes it essential to preserve it in kerosene or naphtha. No separate accounting is made of the metal or its sodium derivatives in United States statistics.

90. Metallic sodium is usually produced by the electrolysis of fused caustic soda (sodium hydroxide) with a nickel anode and iron cathode. Oxygen is liberated at the anode and passes out. Sodium and hydrogen are obtained in the cathode compartment. The sodium deposited on the cathode surface loosens, floats upward, and gathers in an iron cylinder. It is removed with a perforated iron ladle and may be cast into molds. This electrolysis requires over 13,000 kilowatt-hours of energy per short ton metallic sodium.

91. It is possible to manufacture the metal from fused salt with less energy but corrosion difficulties are encountered. A molten lead cathode is sometimes used, requiring a subsequent electrolysis of the sodium-lead alloy produced. Many other types of cells have been invented for this electrolysis but few are in commercial use.

92. When small pieces of metallic sodium are subjected to dry air heated to about 300° C., sodium peroxide (Na_2O_2) is formed. Sodium peroxide, when brought in contact with water or dilute acids, decomposes, yielding hydrogen peroxide. Consequently, sodium peroxide is used as a bleaching agent for textiles and straws and also in the refining of oils and fats. It competes with other hydrogen peroxides in the bleach market.

93. Sodium cyanide is made by passing ammonia through melted sodium mixed with charcoal. It competes in gold mining with cyanide made by fusing cyanamide and sodium chloride in an electric arc furnace and cooling the fused mixture quickly. From this calcium cyanide, hydrocyanic acid for fumigation purposes is produced. This acid competes with sodium cyanide fumigating liquors made by the sodium-ammonia process. Metallic sodium also forms an alloy with lead, which alloy is used for the preparation of hydrogen.

94. The Roessler & Hasslacher Chemical Co., then operating as the Niagara Electro-Chemical Co., began the manufacture of sodium by the electrolytic process. They have continued its manufacture for alloys, and also produce its derivatives—sodium peroxide, hydrogen peroxide, and sodium cyanide—at Niagara Falls. Liquid hydrocyanic acid is manufactured by them at El Morete, Calif. This firm reports sodium as having extensive use in organic industries.

95. *Sodium chlorate.*—Sodium chlorate is manufactured by electrolyzing a hot concentrated salt solution, the sodium hydroxide and chlorine generated being allowed to mix, thus producing the chlorate. This chlorate solution is evaporated and crystallized. The chlorate is used in the production of aniline black and other dyes.

95¼. *Sodium hypochlorite.*—Sodium hypochlorite (NaClO) is produced by the electrolysis of a cold solution of sodium chloride as well as by the chlorination of sodium carbonate. It is used as a deodorizer and disinfectant in dairies and ice-cream factories and also as a bleach in laundries and textile plants. In 1927, 23,118 tons of sodium hypochlorite were manufactured for sale by 11 establishments in the United States. While some electrolytic chlorine plants report the manufacture of this chemical, how much of the total production was by electrolysis is not known.

95½. Cells are of soapstone, concrete, or stoneware. They are divided into compartments by glass partitions so arranged that electrolyte enters and leaves each compartment above the electrodes or so that it is circulated over one and under the next electrode. Electrodes may be of graphite or platinum-iridium wire. The yield is 1 ton of active chlorine per 4,800 kilowatt-hours to 6,200 kilowatt-hours of energy in different types of cells.

Magnesium

96. The magnesium industry has undergone rapid changes in the last few years. The successful manufacture of primary magnesium from the magnesium chloride in brine has ended all previous manu-

facture of the metal from the ore magnesite. With cheapened production and the growth of aeronautics which require metals having the specific properties of magnesium, the magnesium industry has advanced rapidly.

HISTORY AND DEVELOPMENT OF THE INDUSTRY

97. *Europe.*—Although pure magnesium metal was produced as early as 1830, it was the production of the metal by electrolysis of a fused bath of anhydrous magnesium chloride by Bunsen in 1852 that laid the foundations for the present magnesium industry. The first industrial trial production was undertaken in France in 1863 by DeVille and Caron. A mixture of anhydrous magnesium chloride and calcium fluoride was reduced to metallic magnesium in closed iron crucibles. Later, Sonstadt in Germany improved this method by a distillation of the impure metal resulting from the reduction of a mixture of magnesium chloride and sodium chloride with sodium.

98. However, the industry, as developed in Germany prior to the war, was based on the occurrence of the salts of magnesium with those of potassium in Germany's extensive potash deposits. A mixture of the magnesium chloride, potassium chloride, and sodium chloride was used as an anhydrous fused electrolyte. The iron cells formed the cathode while carbon anodes were used. The light magnesium floated on the top of the bath and was ladled off.

99. *United States.*—Although an American company produced small amounts of magnesium in the latter part of the nineteenth century, magnesium used in this country prior to the war was imported from Germany. Such metallic magnesium was used for photographic flashlights, fireworks, and for experimental purposes.

100. *World War production.*—The first recorded production in the United States was in 1914 and 1915, at which time 43.75 tons were produced. This was manufactured by the Rumford Metals Co. at Rumford Falls, Maine. The demand for magnesium in military pyrotechnics, tracer bullets, flares, and as a constituent of explosives was the incentive for the establishment of our domestic magnesium industry during the World War.

101. By 1917 five producers were operating and production had reached almost 58 tons. These producers were the American Magnesium Co., at Niagara Falls, N.Y.; the Dow Chemical Co., of Midland, Mich.; the General Electric Co., at Schenectady, N.Y.; the Norton Laboratories Co., at Lockport, N.Y.; and the Rumford Metals Co., at Rumford Falls, Maine. Production reached 142 tons in 1918. With the cessation of hostilities the industry slumped and production ceased at three factories.

102. *Post-war development.*—With the development of aeronautics came a demand for metals with a maximum strength in relation to weight. This demand was first met by the light aluminum alloys containing from 0.025 to 2 percent magnesium. The most striking property of magnesium is its extreme lightness. With a specific gravity of 1.74, it is only two thirds as heavy as aluminum, one fourth as heavy as iron, and one fifth as heavy as copper. By proper alloying and heat treatment, magnesium alloys were obtained with strength and toughness which permitted them to compete successfully with aluminum alloys in airplane construction.

103. By 1923 the demand for the metal in airplane construction led to an active resumption of activities. Production has increased steadily since that time until in 1929 almost 615 tons were produced. This represented a 155 percent increase in a year. From the close of the war until May 1927 this production came from two companies. The American Magnesium Corporation (a subsidiary of the Aluminum Co., of America) produced the metal at Niagara Falls from calcined magnesite from Washington State or Austria. The Dow Chemical Co. produced magnesium from magnesium chloride recovered as a byproduct of its salt wells at Midland. The cholride process, employing what had been waste material, proved to be so much less expensive than the oxide process used on calcined magnesite that the American Magnesium Corporation closed its factory in May 1927. All equipment for partial fabrication of the metal and for manufacturing the extruded bearings, alloys, castings, forgings, sheet, wire, and powder was moved from Niagara Falls to the plant of the United States Aluminum Co. (leased by the Aluminum Co. of America) at Cleveland, Ohio, where these magnesium shapes are now produced rom metal manufactured by the Dow Chemical Co.

PRESENT PRODUCTION

104. *United States.*—The Dow Chemical Co. of Michigan is, at present, the sole producer of magnesium in the United States, and importations of magnesium from Germany have decreased markedly.

105. A company was formed in 1929 for the purpose of producing magnesium, although no production was reported for this year. This was the United States Metallic Magnesium Co. which proposed to manufacture magnesium metal from dolomite deposits near Logan, Utah.

106. Prior to 1927 the fabrication of the magnesium into semifinished and finished forms was done almost entirely by the two plants manufacturing the metal. Consequently, all statistics prior to 1927 include the ingot and its remanufacture. Since 1927 virgin ingot only is included in production figures and statistics for the manufactured magnesium are listed separately.

107. Table V indicates clearly the very real increase in the domestic production of magnesium. It also shows the decreasing cost of the metal that has accompanied this increased consumption; and in January 1931 ingot magnesium was quoted at $0.48 per pound.

TABLE V.—*Domestic metallic magnesium consumed in United States*[1]

[Virgin ingot only reported after 1927; prior to that year all forms sold were included]

Year	Consumption	Average ingot price per pound	Year	Consumption	Average ingot price per pound
	Pounds			*Pounds*	
1915	87,500	$5.00	1925	245,000	$0.86
1920	123,770	1.60	1926	322,650	.80
1921	48,000	1.30	1927	311,600	.90
1922	[2] 60,000	1.60	1928	521,075	.875
1923	[2] 125,000	1.25	1929	1,329,669	.862
1924	128,000	1.10			

[1] U.S. Bureau of Mines furnishes production figures and estimates. [2] Estimated.

108. *Europe.*—Magnesium production in Europe is also confined to a few companies. The I. G. Farbenindustrie A. G. controls German production, which is unofficially reported to total 2,000 tons annually. The metal is being used not only in German aircraft, but in automobile busses and trucks. The ingot is also sold to French and Italian fabricators. It is believed that the metal is still produced by the Magnesium Co. at Wolverhampton, England. A new factory in Switzerland was in course of erection in 1930.

109. *Imports.*—As a direct result of the Tariff Act of 1922 imports of magnesium decreased considerably and from 1924 to 1928 constituted only about 5 percent of the domestic consumption. In 1929 imports dropped to less than three tenths of 1 percent of total magnesium available in the United States. There were no changes in the duty on magnesium included in the Tariff Act of 1930, the duty being $0.40 per pound on metallic magnesium and scrap; $0.40 per pound on magnesium content plus 20 percent of the foreign market value on manufactured forms, including alloys.

TABLE VI.—*Magnesium imported to United States during last 10 years* [1]

Year	Pounds	Value		Year	Pounds	Value	
		Total	Average			Total	Average
1920	29,275	$25,055	$0.85	1925	8,326	$7,070	$0.85
1921	49,913	30,592	.77	1926	10,117	4,750	.47
1922	182,939	54,448	.30	1927	7,131	8,402	1.18
1923	13,974	11,576	.83	1928	12,039	11,890	.99
1924	8,738	6,561	.75	1929	3,490	6,539	1.87

[1] U.S. Bureau of Foreign and Domestic Commerce.

110. Table VII shows that none of the imported magnesium in 1929 was virgin ingot. Powdered magnesium was the only form in which imports were large enough to attract attention.

TABLE VII.—*Magnesium products imported to United States in 1928 and 1929* [1]

Class	1928			1929		
	Pounds	Value		Pounds	Value	
		Total	Average		Total	Average
Alloys (magnesium content)	20	$57	$2.85	52	$147	$2.83
Magnesium (metallic and scrap)	1,000	1,048	1.05			
Powder (magnesium content)	10,133	9,443	.93	2,951	5,351	1.81
Sheets, tubing, ribbons, wire, and all other	886	1,342	1.52	487	1,041	2.14
Total	12,039	11,890	.99	3,490	6,539	1.87

[1] U.S. Bureau of Foreign and Domestic Commerce.

SOURCES OF RAW MATERIAL

111. *Brine.*—As is indicated on page 283 of this report, oceanic salts contain about 11 percent magnesium chloride and 5 percent magnesium sulphate, while oceanic water contains about one eighth percent magnesium. The bittern from salt crystallization is made up

very largely of crystalline magnesium chloride ($MgCl_26H_2O$). The Great Salt Lake salt contains 2 percent magnesium or its waters hold 0.56 percent. The anhydrous brine in the Saginaw Valley of Michigan holds from 3 percent to 7.5 percent magnesium. At Syracuse, N.Y., Pomeroy, Ohio, and at Hartford, W.Va., some magnesium-bearing bitterns are also found. There is a dry-lake deposit of pure magnesium sulphate near Oroville, Wash.

112. Not all saline deposits have been analyzed for their magnesium content. It is likely, therefore, that salt beds in Utah and in other localities also contain magnesium chloride.

113. The only commercial producers of magnesium chloride in the United States in 1929 were the Dow Chemical Co., of Michigan, and the California Chemical Corporation with plants at Newark, San Mateo, and at Chule Vista on San Francisco Bay. Natural magnesium sulphate was produced only by the Dow Chemical Co.

114. The most famous salt-bed deposits are those in the Magdeburg-Halberstadt region of Germany, where the great Stassfurt salt beds contain the magnesium-bearing mineral, carnallite. Magnesium-bearing salt beds are found also in Alsace and in Spain. Imports to the United States totaled 1,646 tons of magnesium chloride and 6,770 tons of magnesium sulphate in 1929.

115. *Magnesite.*—In addition to magnesium contained in brine, large deposits of magnesium ore occur in different combinations. Magnesite, a carbonate of magnesium, is found in Washington State and in California. The Northwest Mangesite Co. operates a mine near Chewelah, Stevens County, Wash. It produces dead-burned or calcined magnesite and small amounts of caustic-calcined magnesite. In California largest operations were in Santa Clara County in 1929. Operations also took place in Stanislaus, Tulare, San Benito, and Monterey Counties. Magnesite has been produced, in other years, in Alameda, Fresno, Merced, Placer, San Bernardino, Sonora, and Tuolumne Counties.

116. War-time estimates placed the California reserves at 750,000 short tons and the Washington reserves at 7,000,000 short tons.

117. What was the old Austria-Hungary was estimated to have 120,000,000 short tons of magnesite. At present, producing countries are Austria, Hungary, Czechoslovakia, Greece, Italy, Spain, Australia, British India, South Africa, and Canada.

118. *Other magnesium ores.*—A large deposit of brucite, a magnesium hydrate, was discovered in 1927 in Nye County, Nev. Experiments are underway to determine its commercial value.

119. Dolomite, a high-content magnesium limestone, is widely distributed in the United States. As yet no metal magnesium has been made from this source of magnesium.

Magnesium Manufacturing Processes

120. As has been stated, only one method of manufacturing magnesium is practiced in the United States today. Although many processes have been patented, they are not actually used commercially.

121. *The chloride process.*—Magnesium chloride occurring in brine may serve as the raw material for this process. Or magnesium chloride may be produced from the mineral magnesite, first by calcining it to magnesium oxide and then by treatment with chlorine gas in the presence of a carbonaceous material.

122. As brine is the raw material source employed in this country its use will be described first.

123. *Brine separation.*—Natural brine, as it is pumped from Michigan wells in the Saginaw region, contains 3 percent magnesium chloride, 0.15 percent bromine, 14 percent sodium chloride, and 9 percent calcium chloride.

124. *Bromine production.*—Bromine is first removed. The brine is passed through electrolytic cells which oxidize the brine and thereby free its bromine content. These cells are placed above blowing-out towers so that the bromine-laden corrosive brine flows by gravity to these towers. In passing down the towers it comes in contact with air currents that remove the bromine by blowing out. This bromine-laden air passes over an alkali solution or iron, which serves to extract the bromine from the air. Bromine is used in the dye industry, in organic synthesis, for the production of medicinals, and for other chemical purposes.[93]

125. Brine impurities are precipitated and separated out in continuous thickeners and sedimentation tanks. The remaining liquor is then evaporated until the sodium chloride has crystallized. This is used for the electrolytic production of chlorine and caustic soda which are, in turn, combined with other basic chemicals to produce a number of compounds. The calcium chloride is then separated from the magnesium chloride by fractional crystallization.

126. *Dehydration of magnesium chloride.*—The remaining product contains 6 molecules of water. Four are removed by air-drying on the counter current principle. The last 2 molecules of water must be removed in an atmosphere of hydrochloric acid gas to prevent the formation of magnesium oxide.

127. *Electrolysis of dehydrated magnesium chloride.*—Rectangular cast-steel pots serve as the cells as well as the cathodes in the electrolysis of dehydrated-magnesium chloride. The anode is built up of graphite bars. The dehydrated-magnesium chloride is fed into the cells as finished metal is removed, the entire operation being continuous. At the Dow chemical plant power consumption is reduced by the use of exterior furnace heat to maintain proper cell temperature and by the addition of sodium chloride to reduce the melting point and increase the conductivity of the bath.

128. The magnesium metal formed by the electrolytic decomposition of magnesium chloride is lighter than the bath and floats while the chlorine is liberated at the anode. The metal is ladled out of the cell, while the chlorine is recovered. The magnesium metal is 99.9+ percent pure so that subsequent refining is unnecessary.[94]

129. Cells as now used can hold several tons of molten cell bath. The theoretical decomposition voltage is 3.25 volts although 5 or more volts are required in practice. The average current is 2,200 amperes or 16 amperes per square inch anode surface. A ton of metal requires approximately 16,000 kilowatt hours of current for its electrolysis.

The Chemical Co., which utilizes all constituents of its brine in the manufacture of metallic magnesium and an interlocking chain of valuable chemicals, has a power plant with a generating capacity of 38,000 kilowatts.

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[93] Dow, Herbert H. Brine and Bromine Plant Development at Midland, Industrial and Engineering Chemistry, February 1930.

[94] Gann, John A. The Magnesium Industry. Industrial and Engineering Chemistry, vol. 22, July 1930.

130. *Magnesite as a raw material.*—In England, metallic magnesium is manufactured from calcined magnesite, the required chlorine being a byproduct of the process.

131. *Preparation of electrolyte.*—By suspending the calcined magnesite in water, milk of magnesium is formed. This is scrubbed with chlorine gas in a series of scrubbing towers. The chlorine is absorbed by the magnesium, the resulting solution containing magnesium chloride and magnesium chlorate in the proportion of 5 to 1. Upon evaporation and cooling, half the magnesium chloride content crystallizes.

132. These crystals of magnesium chloride are put through an extractor, are exposed to dry air currents and are lastly treated by dry hydrochloric acid gas to remove all water. The resulting product is 99 percent magnesium chloride.

133. As in the brine process, a little sodium or potassium chloride must be added to the magnesium chloride to produce the electrolyte, as the melting of anhydrous magnesium chloride, 750° C., is above the economical electrolysis temperature.

134. *Electrolysis.*—The English company uses two separate cells for the electrolysis. The first cell is charged with a few inches of molten lead which forms the cathode. A graphite anode is used. The electrolyte and the lead cathode are kept in constant circulation. An alloy of magnesium lead is formed which serves as the anode in the second cell. In this cell the cathode is made up of steel rods, and the electrolyte is magnesium chloride. The chlorine liberated from the cells is used in the milk of magnesia scrubbing towers. About 8.5 kilowatt-hours of electricity are required per pound of magnesium produced, or 17,000 kilowatt-hours per ton.

135. *Byproducts.*—By adding potassium chloride to the mother liquors after the magnesium chloride has crystallized out, potassium chloride is obtained. After washing and recrystallizing, this is available for the manufacture of explosives, matches, or pyrotechnics.

136. *The magnesium oxide process.*—Although this process has been superseded by the chlorine process on brines, it is of interest here because the magnesite deposits of the Pacific coast or Austria furnished the raw material.

137. The electrolyte consisted of equal parts of magnesium and barium fluorides to which some sodium fluoride was added. A steel box, having cast-iron cathodes extending longitudinally through its base and a graphite anode in the center of the top, served as the cell. The magnesium oxide, or calcined magnesite, was added to the bath near the anodes at frequent intervals. As the liquid magnesium separated out and ascended, it was caught in collecting hoods placed at the top of the cell. From these it was drawn off as desired. The metal was then refined by volatilization and condensation, or by remelting with a flux. One ton metal required 1.7 tons calcined magnesite.

138. These cells required from 9,000 to 16,000 amperes when operated at 9 to 16 volts. Current efficiency averaged but 50 percent, while power efficiency was but 10 percent. It is obvious that the efficiency of the oxide process was low, although it had low raw material and labor costs.[95]

[95] Harvey, W. G., vice president, American Magnesium Corporation. Production of Metallic Magnesium from Fused Salts. Transactions of the American Electrochemical Society, vol. XLVII ,1925.

FABRICATION OF METAL

139. *Melting and casting magnesium.*—Molten magnesium oxidizes rapidly near or above its melting point. Its extreme lightness is responsible for a tendency to trap air and gases. Consequently, pure magnesium melting operations must exclude air. This is usually done by a flux consisting of an anhydrous mixture of magnesium and sodium chlorides such as is used as the electrolytic bath; this flux also prevents nonmetallic contaminations.

140. The magnesium is melted in iron or steel pots. It is dipped from the pots by means of ladles equipped with skimming lips which part the protecting flux film. It is poured into permanent molds; all castings except green sand molds may be used.

141. The commercial magnesium-base alloys have lower melting points than the pure metal and, therefore, do not oxidize as readily. Aluminum and zinc alloys are made by adding fixed amounts of the solid metals to liquid magnesium, or all metals desired in the alloy may be melted and charged together.

142. *Commercial forms.*—Both magnesium and certain magnesium alloys can be readily rolled hot and, to some extent, cold. In general, the rolling, extrusion, drawing, and forging processes employed for other metals may be used for magnesium although the details of operation vary. It is easily machined with the usual carbon-steel tools.

143. Substantially pure magnesium is furnished the trade in the form of sticks, ingots, blocks, rods, bars, tubes, sheets, plates, wire, ribbon, powder, and foil. More than two thirds, however, is sold as ingot.

144. Table VIII indicates the form in which magnesium, other than ingot magnesium, is marketed. The increase in magnesium castings is outstanding.

TABLE VIII.—*Marketed production of manufactured magnesium (other than ingot)*[1]

Product	1926	1927	1928	1929
	Pounds	*Pounds*	*Pounds*	*Pounds*
Total pounds sold	85, 895	114, 290	137, 232	196, 321
Alloys	6, 862	11, 835	16, 210	13, 145
Castings	36, 936	31, 992	55, 861	116, 350
Powder	31, 551	31, 658	27, 294	35, 943
Wire and ribbon	5, 662	4, 748	7, 695	7, 736
Rod and tubing	614	1, 191	719	1, 864
Sheet	4, 198	10, 883	8, 425	8, 512
Shavings	72	42	810	720
Other	------	21, 941	20, 218	12, 051

[1] U.S. Bureau of Mines.

MAGNESIUM CONSUMPTION

145. *Pure magnesium.*—In powdered form, magnesium is used for flashlight work and in pyrotechnics, as it burns with a bright light when exposed to air in finely divided condition. Barium nitrate, potassium chlorate, or other oxidizers are added to it, however, to insure rapid and complete combustion. It was this demand for magnesium for military signals, flares, tracer bullets, and other pyrotechnics that led to the intitial establishment of the industry in the United States during the World War.

146. Its deoxidizing characteristics make it useful as a deoxidizer of other metals and of salts. It is employed in the nickel and monel metal industry particularly, where it is placed in an inverted cup underneath the molten metal in the furnaces. Some is used for scavenging scrap brass and zinc-base die-casting alloys, and to remove oxygen from chlorates and other salts.

147. Magnesium, alone or alloyed with copper, aids the synthesis of a number of organic chemicals. It is used principally for medicinal and perfumery preparations.

148. Magnesium wire and ribbon find a market in the radio lamp industry where they are used in the degassification of radio tubes. In sheet form it enters into radio rectifiers. As ribbon it can also be used as an insulator in heating appliances. And from sheet magnesium, moving parts of precision instruments, meter disks, chemical balance points, dry-battery elements, and of other delicate mechanisms are made.

149. *Magnesium alloys.*—But the recent growth of the industry is based upon its use in magnesium-base alloys, or alloys containing 90 to 95 percent magnesium. Magnesium alloys with most of the common metals other than iron and chromium. Manganese and zinc are regarded, however, as the best elements for alloying with magnesium to add high mechanical strength, toughness, and corrosion resistance to its extreme lightness. Castings made from such lightweight but strong magnesium alloys are of vital importance in airplane construction where savings in weight are essential. They have been used in such aircraft motor parts as crankcases, oil pans, fuel line fittings, and pistons, and in control levers, instrument housings, superchargers, and in seat frames. Magnesium alloys possess the same value of lightness combined with strength in portable machines and in machine tools that require continuous handling.

150. The principal marketed magnesium alloys are: A magnesium-aluminum-manganese alloy used for castings; a magnesium-manganese alloy having maximum corrosion resistance; an alloy of magnesium-aluminum-copper-manganese and carbon with high thermal properties.

151. An increase in magnesium consumption has been brought about by its use as a minor alloying ingredient of aluminum alloys such as duraluminum. These are manufactured into automobile pistons and other reciprocating engine parts, aircraft engine parts, and into structural parts. (See p. 127 of Power in Aluminum Production.) Unquestionably, every lowering of the price of magnesium nearer to the price of aluminum (about $0.23) increases the competition of the light aluminum alloys with the lighter magnesium alloys.

152. In zinc-base die-casting alloys small amounts of magnesium are used to prevent deterioration of the zinc when exposed to hot, humid atmospheres.

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