TABLET MANUFACTURE.*

BY ROBERT C. WHITE.¹

The story of tablet making is indeed an interesting one. It possesses so many phases.

Medical Viewpoint.—Having been originated for the chief purpose of introducing to the human body medicinal ingredients some naturally think of it from a medical viewpoint. Such questions arise as to whether the various more or less soluble chemicals can be introduced into the body by this means. Some will always argue that medicine in solutions is preferable on account of its much more speedy absorption, while others pay special attention to the keeping quality of the various ingredients, and the convenience with which medicine in tablet form may be conveyed from place to place, or may be carried by the user himself, and how the layman has the medicine in properly subdivided doses for self-medication.

Mechanical Viewpoint.—The man of mechanical tendencies naturally thinks of the manufacture of medicinal tablets as one involving machinery and mechanical appliances for the proper powdering, the proper sub-dividing, moistening, drying, screening and compressing of material into tablet form. The various shapes of tablets, the different weights, the different colors involved, the raised lettering on some, the concave engraving on others; all of these processes are much affected by temperature, humidity, exposure to light, and to foreign particles, etc.

Viewpoint of Pharmaceutical Chemist.—The pharmaceutical chemist will, of course, give attention to the solubility, to the preservation, and keeping qualities of tablets, the color reaction, the deliquescent tablets, the tablets of volatile ingredients, the tablets made for speedy disintegration, for absorption in the stomach, or for absorption in the intestinal tract; the man, in other words, whose viewpoint is controlled by the need of the physician and by the possibility of mechanical adaptations. Thus we find that tablet making involves a wider field than most of us think when first considering the subject.

Origin.—Tablet making was first begun in England about sixty-eight years ago. The writer has never been able to find any very definite information as to who the first tablet maker may have been, but doubtless the idea originated from the association of lozenges and of pills, which were well known before that time. Tablets as originally designed were simply intended to bring together such dry medicinal ingredients as might be compressed into small discs. In fact this new form was improperly called "compressed pill." Pills, as we all know, are really made from the plastic mass, combining the various ingredients.

Difference between Tablets and Pills.—The idea of compressed pills, however, is not as erroneous to-day as it was fifty years ago; because to the great list of simple compressed chemicals, or list of dried chemicals, has since that time been added the combination of granulated masses containing solid or semi-solid vegetable extractives, which during the process of admixture were really plastic matter similar to the original pill masses. In other words, the tablet masses of to-day contain practically all formulae which may be found in pill combination. We

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¹ Pharmaceutical Engineer.

need not here concern ourselves as to whether medicinal ingredients given in tablet or pillular form are best from a physiologic standpoint. This argument will continue to go on for years to come, and the writer's only comment to this discussion is that a competent tablet manufacturer can make practically any tablet from any formula that can be made in pill form which will disintegrate as readily in the fluids of the stomach as will any pill that the best pill maker may turn out. Great objection was at first made, and justly so, to tablets on account of their sparing solubility, the great length of time necessary for disintegration, all of which was readily proved by the fact that many tablets as made during the first thirty years of tablet history would pass through the human system showing as much change as would a glass marble. This, of course, gave great opportunity to the manufacturers who had installed expensive pill machinery, and who had worked out very proper pill formulae, to justly ridicule the idea of compressing dry ingredients into any useful form of medication. Since tablet-making has become a science, however, these objections are by no means sustained, and, as all manufacturers realize, the number of tablets manufactured greatly exceeds that of pills. The retail druggist, of course, has occasion to manufacture pills upon prescription, but practically never finds himself in the position where he has to manufacture tablets, although there are on the market two or three small mechanical devices which will permit the druggist to manufacture tablets of fair accuracy.

TABLET MANUFACTURE.

Upon investigation the writer finds that during his sad connection with the fraternity of the mortar and pestle he has been involved in the manufacture of over thirty-four hundred different formulae for compressed tablets. It is, therefore, obvious that in this paper we can go into no detail giving working formulae for the manufacture of various kinds of tablets. These will, therefore, have to be treated in classes. Suffice it to say no tablets of different formulae are ever the same in every detail of manufacture, but on account of the similarity of some, they may be grouped under similar processes. Tablets as a rule are lenticularshaped discs from four to ten times as wide as they are deep. This shape proves convenient for tablets, either designed to be swallowed or dissolved in the mouth, as they lie comfortably on the tongue. The same ingredients can easily be made in varying weights, thus permitting a wide range of medication according to the age and condition of the patient. The majority of tablets will not disintegrate in the mouth, but can be quickly swallowed, and are thus a most useful form of medication on account of their tastelessness. Tablets as a class possess great keeping qualities. On account of the moisture present in the plastic mass, pills, unless heavily coated, frequently dry out and crack, thus destroying the perfect finish of the pill.

Mechanical Methods.—The three parts of a compressing machine which come in contact with the ingredients to form a tablet are the upper punch, the lower punch, and the die into which these punches are thrust by mechanical means. In other words, the tablet is formed by pressure on the ingredients between two punches restricted in expansion by the surrounding die. Standard sizes of punches and corresponding dies are well known in sizes from 1/32 to 1 inch in diameter, and when the tablets do not require coating they are usually made standard cup, *i. e.*, of a known concavity. For coating a deeper cup is used as this gives the tablet a more spherical outline, which permits of the tablets rolling more uniformly in the coating basin. Punches and dies can be so made as to give almost any shaped tablet. Many



Fig. I .--- Special Shape Punches and Dies.

manufacturers of certain specialties often adopt "freak" shapes for their tablets on account of the individuality their product derives, but many shapes are both useful and ingenious, to wit, the coffin-shaped bichloride tablet, the life-buoy shaped "Life Saver" mints and many others. It would be well to fix firmly in mind at the present time that fine powders cannot be compressed into tablets. This is for the simple reason that the air which feeds into the die with the ingredients, on account of the tight fit of the punches and the die, cannot escape when the blow is struck; whereas the use of granules permits the air to escape more gradually on account of the interstices formed due to these irregular particles. This rule, therefore, gives the manufacturer considerable difficulty in that, in the ma-

jority of cases, granules have to be built up from the fine powdered ingredients involved. This necessitates the use of considerable machinery and an excipient or binder.

Simple Salts for Granulation.—There are certain chemicals that in their original form can be readily used for tablet making. These include the iodides, bromides and chlorides, of such salts as potassium, sodium and ammonium. Potassium bicarbonate, potassium permanganate, sodium phosphate, zinc sulphate, and even sugar can be compressed in this simple way, if they are reduced to a size which will pass through a number twenty screen, but not through a number thirty. This eliminates fine powder. These salts can be readily bought in crystal form, and ground to sizes between a twenty- and thirty-granule, which can be



Fig. II -- Sifter.

fed direct into the hopper of a compressing machine. It is necessary, however, to exercise care in the case of some of these single salts that all surface moisture be removed though none of the water of crystallization must be disturbed, otherwise the chemical content of the tablet will vary. Mixed ingredients give a great deal more trouble not only from the incompatibility involved, but from the mechanical manipulation necessary for the uniform distribution of the various ingredients through the entire mass.

Mixed Salts.—It might appear at this stage that if we can compress a simple granulated salt, we should be able to mix two or more of these salts together in the proper proportions, and make from them uniform tablets. This is not the case in ground crystals as it will be found that all crystals fracture differently. Thus in ground salts we will find that one will contain more fine particles than will another, and these will not feed evenly into the die. This will be readily demonstrated if you will examine a tablet the appearance of which is *mottled*. On account of containing certain colored ingredients, one side of this tablet will be prominently mottled while the other side of this tablet will be colored more evenly. This is due to the fact that although only a ninetieth or hundredth of a second is necessary for the compression of the tablet the fine particles fall onto the surface of the lower punch while the coarser particles remain on the top. If you will stand by a tablet machine, and lift the tablet as it comes from the die plate, you will see that the lower side of a tablet is always smoother, if colored ingredients are involved, than the upper. If practiced on dry mixed ingredients the results would unquestionably be that some tablets would contain more of some ingredients than others, so that in the vast majority of cases it is necessary for the manufacturer to reduce all the ingredients to an impalpable powder, place these in a



Fig. III.-Mixer.

heavy mixer, and by the use of various excipients reduce the combination to a uniform mass.

Mixing of the Mass.—It will be here found that just as in making fluid mixtures the various ingredients must be fed into the mass in proper sequence with due regard to both chemical and physical reaction. In all cases before putting ingredients into a mixture for the making of a mass all potent remedies should be very carefully forced through a silk sieve, and to accomplish this, in the majority of cases, a ball mill should be used for the powdering of the ingredients.

Mixers.—Many types of mixers are necessary for the purpose of the manufacturer. They come chiefly in two classes, those for mixing dry powders, which usually have screening apparatus attached, and those for mixing heavy plastic masses. An entire book could very easily be written on the subject of mixers. Those for the plastic mass of necessity have very powerful drives, and require very strong transmission. Those with a single set of beaters, or blades, are not sufficiently strong for the sticky masses, as the mass will collect on the blades and turn over in its entirety without doing any real mixing. For this reason those having two sets of kneading blades are preferable, as these throw the mass into the center, and then crush it out to the sides of the mixer. Many of these mixers are so constructed that these blades can be reversed so that while at one time they will force the mass into the center and downward, when reversed they operate the mass from the center and crush it against the sides upwards. Many of these are steamjacketed, thus permitting the mass to be kept soft and plastic, while the mixer is being operated, but letting the mass become nearly dry when cool. This, of course, saves just so much drying after the mass has been placed on trays.



Fig. IV.-Sifter and Mixer.

Excipients.—Water, alcohol, glucose, gelatin, starch paste or jelly, honey, or acacia may be used for excipients. Gelatin and acacia should always be reduced to a jelly or a fluid consistency. At times a manufacturer finds it necessary on account of the physical properties to use ten, twenty, thirty or forty percent gelatin or acacia solution. In some cases it has been found that these should be used warm, in others quite hot, still again certain masses work perfectly with cold solutions. Powdered gelatin or powdered tragacanth should never be used, for slowly disintegrating tablets will result. Some manufacturers, however, find

it expedient to use a small proportion of powdered acacia with the dry granulation after it has reached the compressing machine, if the tablets are not binding properly. This practice should be discouraged. In a great many tablet masses con-



Fig. V.-Ball Mill.

taining vegetable extracts it will be found that these extracts can be used either as a solid extract or in more dilute form, which eliminates the need for other excipients. Glycerin should never be used, as glycerin will not dry out, and forms a gummy tablet, which cannot be coated successfully, cannot carry lettering, and will invariably stick either to the die, or to the punch.

Drying.—The masses, when removed from the mixers, are then subjected to drying. They are broken into coarse particles, and usually spread on sheets of glass, aluminum trays, paper with a properly sized surface, or on cheese cloth, and are then subjected to varying temperatures for different lengths of time. Many types of drying closets are used. For years the most popular was the ordinary

closet, arranged in series, heated by steam coils in the back, having an opening near the base, and an opening for the escape of the evaporated moisture at the top. Trays, the surfaces of which were covered with canton flannel, were used in these dryers; the trays being put into the dryers singly, or stacked up on racks, which were then pushed into the dryer. There are objectionable features to this kind of dryer. In the first place the heat cannot be distributed evenly, but will be found more intense at the back of the dryer. This was partially overcome by the continual turning of the trays but the method was wasteful in time and There was no proper air control and most of the moisture evaporated labor. depended entirely on the ascension of the hot air. These air currents often carried foreign particles into the dryer and contaminated the product. Later came development involving the use of mechanical blowers, the heating of the air in outside chambers, and the uniform distribution of the hot air by means of properly arranged flues. This evolution has at last brought us up to very carefully arranged dryers, which show very ingenious steam control, and also the control of the air current, obviating the need for the removal of trays after the dryer had been closed. These dryers are now being manufactured of entirely fireproof material, which can be kept perfectly clean, and do not allow of any contamination even though different products be placed in the same dryer at the same time. There will always be, however, certain products which on account of their very volatile nature must be dried at a very low temperature. The ideal way for drying all granulation is by the use of a vacuum dryer. This aids in retarding the escape of volatile ingredients, and the discoloration of the various salts and extracts

present. Suction pumps have been made so nearly perfect in the last few years that a working vacuum of 28 or 29 inches is not exceptional, and of course no contamination under these conditions is possible. A vacuum system, however, is much more expensive in cost and operation than the air dryer, but just as we must reduce many fluidextracts in vacuum stills rather than in the ordinary evaporating pans, the manufacturer who seeks to turn out a first-class product will be perfectly



Fig. VI.-Double Blade Mixer.

willing to maintain several of these vacuum dryers. They are also absolutely necessary in making granulation for effervescent tablets, where it is desirable to retain a full degree of effervescence. A more speedy cessation of reaction is possible in the case of effervescent ingredients when handled *in vacuo* instead of in the ordinary hot air dryer. Unquestionably in years past the usefulness of tablets was impaired on account of the granulations being virtually baked. The length of time necessary for drying and the proper temperature has been worked out to a very fine degree. It will be readily seen that masses containing volatile oils, camphor, chloral hydrate, the salicylates, caffeine, salol, and like ingredients cannot be subjected to a high degree of temperature. In fact many of these have for years been air dried, meaning they have been dried at normal temperature. In all cases it is absolutely necessary to have granulations containing these ingredients examined before they are compressed into tablets, in order that the manufacturer may be sure that the full amounts of all ingredients claimed are present. In fact some of these volatile ingredients are mixed with the balance of the prepared ingredients immediately before placing them on the compressing machines, and are not incorporated in the original mass, and thus forced to go through exposure to heat. The writer has often seen crystals of benzoic acid, caffein, and like materials sublimed on the "sight glass" on the face of a vacuum dryer, where the temperature was only 100° F. This shows the great care that must be exercised in drying, not so much from the standpoint of wastefulness, as for the need of turning out a finished product containing 100 percent of all the ingredients incorporated. These granulations, when dried, must be very carefully screened.

Screening.—A great many well-trained pharmacists still have the idea that a number 20 granulation or powder means anything that will pass through a screen



Fig. VII.---Rack for Trays.

having twenty meshes to the linear inch. This idea if carried into practice with the manufacturer would play havoc with his methods. To him a number 20 granulation means that which will go through a number 20 screen, but will remain on the surface of a number thirty. The adherence to this rule is necessary, as has been shown in the foregoing paragraphs. With the developing of certain improved screening apparatus the manufacturer is fast getting away from the old hand methods which were extravagant in time and labor, and were also objectionable on account of the possibility of contamination from the hands of the operators.

Very clever apparatus has been devised, which in the case of different size granules requires only the changing of the piece of wire screening, which can be done in a few moments.

Protection from Exposure.—The granulation must be protected during its entire progress from all contamination with outside sources. It should be quickly fed into the hopper of the machine and the hopper immediately covered. Naturally, where power transmission is so largely involved as in tablet making, there is continually the possibility of contamination from dust due to the moving shafting, pullies, belts, and also lubricating oils necessary for all moving parts.

Suction Systems.—The dust involved, however, is removed in most wellordered plants by having a suction system terminating in hoods in the close proximity of the tablet machines. How very necessary these suction systems are, has been oft-times demonstrated by tieing a canton flannel sack over the exhaust end of this system and examining the dust which is found collected there. This examination shows the manufacturer why he loses from two to eight percent of his ingredients in the manufacture of tablets. In some cases where continued runs of only one tablet are made, the air from the exhaust end of this suction system is forced through water, and the trapped material is then recovered from the solution. In the majority of cases it proves best to feed the granulation into the hopper while still slightly warm.

Moisture.—The presence of moisture makes the tablet stick to the dies or punches and in some cases precludes all possibility of operating the presses on certain types of tablets during the humid months of July and August in these localities, unless air, dried over calcium chloride, is forced into the room containing the presses.

Polishing Dies and Punches.—Now naturally arises the question as to how to prevent the more or less developed tendency to stick. In the first place the surfaces of dies and punches are polished to a mirror-like surface, and these have to be repolished frequently. It is usually done by simple devices assisted by the use of the finest emery obtainable, worked into a paste-like consistence with glycerin. Some ingredients wear the surfaces rough more quickly than do others. For instance, the punches and dies employed in the making of a tablet containing five grains of Blaud's Mass will show wear under fifty thousand production, whereas the same punches and dies could be used for making a million tablets of aspirin or some of the acetanilid group without showing any perceptible wear. This wear also affects the appearance of the tablet. It will often be found that edges, and rings by some spoken of as "wings," form on tablets due to the fact that there may be a worn condition of the punch or die which permits a slight amount of the tablet



Fig. VIII.-Gordon Dryer.

ingredient to squeeze up between the die and punch. This is objectionable, not only on account of appearance, but these rims will rub off the tablets after they are packed in bottles, and will make the tablets and container dusty. The only solution for this difficulty is a new set of dies and punches. A tablet from a highly polished surface will in a great many cases show a glass-like finish on top, bottom and edge. It is impossible, however, to give some tablets the fine glass-like appearance which is to be found on others.

Moist Appearance.—If you will stop to think you will remember that you have never seen a shiny aspirin tablet, nor have you ever seen a shiny quinine tablet, or an acetanilid tablet having a high-surfaced appearance. These tablets some manufacturers speak of as the "moist group," because they always have a dull, moist appearance. You will also remember that the bottle containing these tablets had always a blurred appearance due to the fact that no amount of blowing will remove the dust from the surface of such tablets. On the other hand, there are thousands of tablets which could be put into a bottle and shaken for a long period of time, as is the case in the shipping of them all over the world, and the tablets will still possess the glass-like surface and the bottle will show no murkiness. Sticking.—It will be found that the first mentioned, or moist tablets, give the greatest difficulty on account of their tendency to stick on the punch. This difficulty is controlled by the addition of a small quantity of talcum powder. The tablets often stick to the punch, which accounts for the concavity sometimes found on the surface of the tablets, due to particles adhering to the punch. The term used for this by the manufacturer is "picking," which means that the punch picks a small surface out of the tablet. If the press be operated at this

stage the hollow in the surface of the tablet will grow larger with each tablet made, as a slight particle of each succeeding tablet will stick to the original speck which has bound itself to the punch. Talcum will obviate this, and is used in varying amounts up to $\frac{1}{8}$ grain to the tablet, according to the size of the tablet and the requirements which the manufacturer maintains for his product. Talcum should never be used unless



Fig. IX.—Gordon Dryer.

absolutely necessary, and then only in the smallest amount which will give the desired results. No conscientious manufacturer would want to put talcum in his tablets if he could possibly avoid it.

Edges.—Now for the edge, or side, of the tablet. Talcum is of no use in this case. It must be remembered that the blow on the material is made by the two punches, whereas the die fulfills a two-fold function. It prevents side-way ex-



Fig. X .- Oscillating Sifter.

pansion of the material under pressure, and it is also the cylinder through which the tablet is forced when the punch lifts it to the surface for its speedy exit from the die plate. The finest grades of white mineral oil have been found most suitable as the lubricant to prevent sticking to the die. Here you will find again that the surfaces of different tablets vary. The edges of some are bright and hard like glass, while others are dull. You will by this time have drawn the very reasonable conclusion that the dull finished tablets require more mineral oil than do those of bright finish.

Tablets for Solution.—It might be well to mention that in the case of tablets to be used for solutions the manufacturer in some cases resorts to boric acid instead of talcum, or as in the case of lithium tablets he may use lithium benzoate, which has proven very helpful. Oil should never be used in these tablets as it will come to the surface of the water when dissolved and present an unsightly appearance

varying in degree with the amount of oil used. This is why all such tablets are usually made of large diameter as they present less surface to the sides of the dies, and consequently bind less.

Disintegration.—As was mentioned in an earlier part of this article the disintegration of compressed tablets is a matter of great moment. Certainly the excipients used and the quality of extractives incorporated, together with the pressure exerted on the compressing machine, play a very positive part in the later disintegration of the tablet. For many years all kinds of experiments were made with tablets of different formulae in order to find what proportions of these various influencing agents would give the speediest disintegration. Manufacturers very carefully treasured formulae of tablets that gave a quickly disintegrating tablet. In fact the entire manufacturing business for years was surrounded by a cloud of great secrecy. In these days of research, however, it would seem to the writer that there is absolutely no need for the reserve that is still practiced regarding methods and formulae. The great disintegrating agent which stands far above all others is potato starch. One-sixteenth or one-eighth of a grain of potato starch will permit



Fig. XI.

Fig. XII. Tablet Presses.

Fig. XIII.

of practically any tablet disintegrating quickly when placed in a solution. The usual test method in the manufacturing laboratory is simply to drop the tablets into water and in the operator's terms "see how soon they break up." Potato starch swells so rapidly, that in nearly all cases tablets will rupture as soon as they are dropped into water. The potato starch should, of course, be added to the dry granulation immediately before placing it in the hopper of the compressing machine.

Lettering of Tablets.—An interesting feature at this stage is the lettering, or as the manufacturer would say, the "monograming of tablets." As a rule where lettering is to be done on tablets a number 30 granule is resorted to, as the coarse particles will not fill evenly into the curves of the engraving. Some ingredients are better adapted to raised letters, while it has been found that with others the lettering should be indented. On account of the great tendency to sticking, no manufacturer would want to use lettering on a tablet unless he felt that considerable value was to be obtained from his having each tablet bear his special mark or design.

Compressing Machines.—It might be interesting to note that the blow on most of our large size compressing machines registers a pressure of from four to six tons. There are several types of machines on the market. The single press, which refers to the one making a single tablet at each revolution, is the most used.

These machines maintain an output of from forty to fifty thousand per day. Then we have the large multiples, which are practically of the same design, only being built much larger, and heavier, and which carry from two to twelve corresponding holes in a single die, thus making from two to twelve tablets at a revolution, or from two to twelve times as many tablets in a day. These multiple presses in later years have largely given way to the rotary press, a press which contains fifteen punches fixed in a rotating collar, having corresponding dies beneath, which punches are independently operated when they come under a deep-seated roller which forces them down. These rotary machines, however, are only applicable where large runs of tablets are to be made, as the difficulties and time involved in changing so many dies and punches are considerable. Multiple arrangement is also found in rotary machines. Within the last two years it has been found possible to



Fig. XIV.-Multiple Rotary Press.

make a rotary machine of sufficient strength to carry three punches to each die, thus multiplying the output of the machine by three. These machines will then give an output of from eleven to twelve hundred tablets per minute. For long runs of five million or more tablets, these machines work admirably, but are quite expensive, and except in the case of the largest manufacturers cannot be economically used. It is the aim, of course, of every manufacturer and tablet machine maker to devise a machine that will run as continuously as possible. When you stop to think that the shutting down of a machine like the last mentioned for even five minutes entails a loss in output of six thousand tablets, it will be seen how necessary it is to have the granulation in absolutely proper form supplied to the machine, and the machine in perfect adjustment. Several of the large pharmaceutical houses in the United States have found it necessary for proper development in this direction to manufacture their own machinery, but very satisfactory machines for general purposes may readily be purchased.

As must have been seen from the foregoing, the life of a tablet maker is not a happy one, and it is the firm conviction of the writer that all tablet makers are made, and not born. Successful tablet making requires a full pharmaceutical

Upper and Lower Punches and Die for Multiple Rotary Press.

training, and in addition requires all the mechanical genius of a millwright, a master mechanic, an engineer, an electrician, and moreover requires the perseverance of a Napoleon, and the patience of a Job; coupled with this the power of leadership, and the ability to coerce operators to do work which they dislike; to have their person stained with various colors which would by no means be mistaken for rouge, to breathe frequently everything in the list from valerian to podophyllin, to taste eternally quinine, strychnine, and cascara compounds. to be muzzled oft-times from morn to night like a rabid canine, and to wear goggles which make you look like a deep sea monster. If a man possessing these qualities can be found, he should after twenty-five or thirty years' experience develop into a good tablet manufacturer.

NEED FOR REGULATION OF THE EXPORTATION OF NARCOTIC DRUGS.*

BY A. W. LINTON.¹

Information in regard to the quantities of narcotic drugs exported from the United States is not easily obtainable. The one who studies the published reports of our exports does not find narcotics scheduled as such, since these are included under the general classification of "drugs and chemicals." It is the pur-

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The China Club of Seattle is an organization including in its membership many of the prominent business and professional men of Seattle, and having for its objects the fostering of cordial relations between China and the United States, and the encouragement of commercial intercourse between the two countries. Dr. McKibben, himself a resident of China for many years and deeply interested in everything concerned with the welfare of that country, has collected much material concerning the shipment of narcotics to China, and this was freely placed at the disposal of the author of this paper.

Mention should be made also of an article by Professor John Dewey, of Peking, which appeared in the *New Republic* of December 24, 1919. Professor Dewey is a member of the faculty of the Columbia University and is now an exchange professor at the University of Peking. He is eminently fitted to write on present conditions in China.

Multiple Rotary Press.

