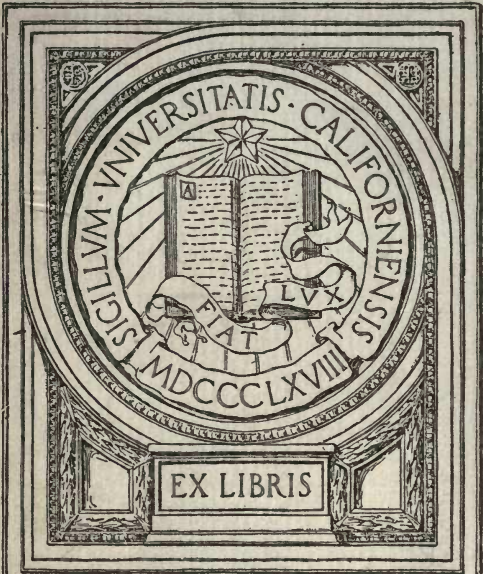
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BLUES AND CARMINES

OF

INDIGO:

A PRACTICAL TREATISE

ON THE

FABRICATION OF EVERY COMMERCIAL PRODUCT DERIVED FROM INDIGO.

BY

FELICIEN CAPRON DE DOLE.

TRANSLATED FROM THE FRENCH, WITH EXTENSIVE AND IMPORTANT ADDITIONS,

BY

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EDITOR'S PREFACE.

We designed, when commencing the translation of this book, to present it to the public without material change. We found, however, in the prosecution of our labor, that the French work was too incomplete to be of much utility for many for whom it was intended, indeed very few would have been benefited by its publication. We have endeavored, therefore, by additions and changes, to render it a practical work not only for manufacturers of carmine, but also for dyers, calico printers, manufacturers of blues and colors, wholesale druggists, etc. etc.

The French author in a few chapters, treats only of the fabrication of the different blues and carmines; we have added a complete chemical history of indigo, such as its culture, its varieties, chemical properties, the action of different acids, the preparation of the most important vats and their uses in the art of dyeing. We close the work with a complete treatise on the commercial assays of the different kinds of indigo. This paper, never before published, is the result of our own experiments during an entire year devoted to the subject while in the laboratory of the Manufacture Impériale des Gobelins. The traders in indigo will be benefited by some of the assays which give the best methods of analyzing indigo. We also give the composition of thirty-eight of the most important varieties. We believe these additions will make the present the most complete work on the subject published in this country, and will encourage the fabrication of a substance so much employed by dyers and calico printers, and which we have been obliged to obtain from importers, and often far from being pure.

THE EDITOR.

NEW LEBANON, N. Y.,
July 6, 1863.

AUTHOR'S INTRODUCTION.

THE indigo found in commerce comes from Egypt, Madagascar, and the East Indies; it is a solid matter, insoluble in water, and soluble in sulphuric acid.

There are several kinds of indigo, the principal of which are the Java, Guatemala, Bengalis, etc. Preference is to be given to the one which has a violet appearance.

It is only since about the end of the last century that indigo has been manufactured, so as to be employed advantageously in giving an azure color to linen and woven fabrics.

The author of this discovery, as useful as it is important, is unknown, and for that reason, several manufacturers have taken to themselves the credit of the invention.

Before the use of indigo, blues manufactured in the form of balls, the raw material of which was Spanish or Troyes chalk, received an azure color by addition of Prussian blue. The azure produced by Prussian blue damaged the linen and woven fabrics by reason of the sulphate of iron that is contained in that blue, which soon becomes of a greenish color; then occurred the discovery of the carmine of indigo used for dyeing, and for the impression of cloth, making the base of the stones, cakes, pastils, blues, etc., of the *Belard's blues*, and of the soluble blues now circulating in the trade.

There are three kinds of blues to be distinguished which are used to give an azure color to linen and woven fabrics.

They are, 1st, the liquid blues, which are nothing else but a sulphuric solution of indigo mixed with water. 2d. The blues in balls, which are also the sulphuric solution of indigo triturated with Spanish chalk. 3. And the blues having for basis the carmine of indigo to which is mixed some fecula, gum in powder, and some sulphate of potash, the last substance only for the soluble blue.

Formerly in the country the use of the different kinds of blues was unknown, but it is no longer the case, they are now employed in small as well as in large families, and have become altogether indispensable; no other matter can take their place, since they alone give to linen and woven fabrics, without damaging them, a light sky blue color, and the appearance of newness to those which are old.

Raw indigo can take the place of carmine, and this is yet employed by many dyers, calico printers, and manufacturers of paper-hanging, probably because they do not know the differences in dyeing or printing between carmine and raw indigo. There is nevertheless a great difference between them in uniting, the dyeing or printing done with the raw indigo always turns green by reason of the green matter contained in the commercial indigo, it is not so with that which is made with the carmine of indigo, which is nearly pure indigo, from which all green, resinous, and oily matters have been taken, it never becomes green, or at least with difficulty, if the carmine has been well prepared.

That mode of fabrication of the blue of carmine of indigo is yet known only to a few persons who make a very advantageous commerce of it; they have never had to fear an opposition, since they have always kept their proceedings secret. They call it a family secret.

Several chemists have indicated processes for obtaining carmine of indigos, but none of them has given one sufficiently simple or cheap. Those who have come the nearest are Mr. Lassaigne in his Treatise of Chemistry, and Thillaye in his Manual of the Manufacture of Chemical Products. Therefore, all processes indicated to this day are very complicated, and too costly to be executed by persons of small means. They have never spoken of the *stones*, *cakes* and *pastils blues*, and the *Belard* and *liquid blues*, or of the *sky* or *soluble blue.*

Hence, in the collection already so numerous of the manuals intended to assist in the arts and sciences, that of the blues and carmine of indigo is not found. In order to supply this deficiency for the interest of the public, particularly for that of commerce, I have made numerous chemical experiments which have led me to discover a mode of fabrication, simple and economical, that may be executed by persons of every class, that is to say, by which the small as well as the large family will be able to manufacture the blues they may use.

This mode of manufacturing is the one explained in this work. The grocers, druggists, dyers, manufacturers of papers, etc., will have a double advantage by manufacturing themselves the blues necessary to their trade, and they will be sure of always having the quality they desire, and will thus gain a hundred per cent. at least. Of this they will be convinced by reading the several chapters of this work.

THE AUTHOR.

CONTENTS.

CHAPTER I.

CHAPTER II.

CHAPTER III.

CHAPTER IV.

CHAPTER V.

CHAPTER VI.

CHAPTER VII.

CHAPTER VIII.

CHAPTER IX.

CHAPTER X.

CHAPTER XI.

CHAPTER XII.

CHAPTER XIII.

CHAPTER XIV.

CHAPTER XV.

CHAPTER XVI.

CHAPTER XVII.

CHAPTER XVIII.

CHAPTER XIX.

CHAPTER XX.

CHAPTER XXI.

CHAPTER XXII.

CHAPTER XXIII.

BLUES AND CARMINES OF INDIGO.

CHAPTER I.

HISTORY OF INDIGO—CULTURE—FABRICATION.

History.

This invaluable dye-stuff, for which no substitute has been yet found, was known to the ancients under the name *indicum*, whence its modern appellation. But the plant from which Indians extracted this fecula is not now used. It seems that this plant is the same that Margraff described, adding that the plant is succulent, and when the trunk is broken, it exudes a blue juice. They made indigo from this plant without any other trouble than grinding it, adding water and letting run. The fecula of indigo varies always, not only according to the plants of which it is extracted, but also according to the treatment to which the plant is submitted. They extract from china a blue dye, from the *Tovara* or *persicaria virginiana*. According to Hermann and Linnæus, they extract from one of the *galega species* a blue dye finer than indigo, and Mr. Guettard has observed in the *Mémoires de l'Académie des Sciences*, 1747, that the ribs of the galega are similar to those of the indigo.

Indigo first came into extensive use in Italy, but about the middle of the 16th century, the Dutch began to import and employ it in considerable quantity. Its general introduction into the dye-houses of both England and France was kept back by absurd laws, founded upon an opinion that it was a fugitive substance, and even prejudicial to the fibres of the wool.

They cultivate it under the name of *anil indigofera* and *indigo*, in China, Japan, India, Madagascar, Egypt, and in America. There are several kinds, but in America they count particularly three: the FRANK INDIGO, *indigofera tinctoria*, which is the smallest and produces the indigo of the lowest quality, but as it gives a large quantity it is often preferred. The second kind is the *indigofera disperma*, cultivated in Guatemala; it is more elevated, more ligneous than the above, and gives a better indigo. The third kind is the *indigofera argentea*, or bastard indigo, which is the most ligneous; it gives the finest indigo, but in small proportions.

It is apparent that this plant absorbs as much more foreign matters which are afterwards confounded with the coloring matter, as it is more herbaceous.

CULTURE.

The *frank indigo* of the Antilles grows about three feet high; it requires a very good ground and much care from the cultivation; the ground must be flat, smooth, damp, and rich. The indigo must be planted during damp weather, in March, in holes about one foot apart and 4 inches deep. Negroes who plant the indigo put about ten or twelve seeds in each hole, and cover it slightly with earth. The plant comes up four or five days afterwards, the trunks at first are knotty, full of small branches, always containing several pairs of leaves and terminated with a single one. They are always careful to destroy the bad weeds. The plant is not slow to produce flowers, and is ready to cut in May. They often make four cuts of the same plant during the year, while in South America, they never make more than two, and ordinarily one, the plant being ready to cut for the first time after six months. The product diminishes continually after the first cut, so that it is necessary to renew the plant from seeds every year.

Fabrication.

When indigo gives signs of being ripe (which is ascertained by the tendency the leaves have to break), it is cut at about two inches from the soil during damp weather, and is carried in the *steeping-trough*, which must be done quickly before the plant grows warm. The steeping-trough is a shed of about 19 feet high, without walls, and sustained by posts; they construct three vats, one on the other, at different heights, and near a receiver of water. The first, which is at the basis, is called *Fuller's soaking-trough;* it is disposed in such a way that the water it contains can run out from the shed; it is in this vat of about 44 feet square, built in masonry or in wood, that the plant is carried, and squeezed in until it is the three-quarters full; thus they add water to it till all the plant is entirely covered, and it is kept in this position, to avoid the floating, by loading it with heavy boards. Very soon it begins a very active fermentation, and it forms a great deal of skims; these skims thicken by degrees, and take a violaceous blue color. The disengaged gas is inflammable. The most convenient temperature for the fermentation is about 80°. If you let it to ferment too long, the coloring matter is altered, and if you remove the water too soon, much indigo is lost.

When the foreman ascertains that the fermentation is advanced enough, and that the coloring matter is disposed to separate, they run the liquid into the second vat, which is ordinarily 34 feet square and 13 deep; the liquid is agitated in it for 15 or 20 minutes with the necessary apparatus; and it is by constant practice that the foreman knows the moment to stop the operation, which is when it begins to separate from the liquid some flocks which gives it a curded aspect. When they judge by the blue color that the beating is sufficient, they leave it to settle for two hours. The indigo forms a kind of mud, which deposits in the bottom of the vat; they leave the water to clear, and they pass it in the third vat, called the *settling-vat*, in which the fabrication of indigo is achieved. They then leave the coloring matter to settle in this vat, and draw the liquid part; they add a certain quantity of lime-water to prevent the putrefaction, and absorb the carbonic acid gas.

Then they take out the indigo which is in half fluid consistence, and put it in thick cloth bags, through which the water runs out; the bags are emptied into long square boxes about 2 inches deep; they are cut in square cakes, and dried in the air to the shade.

The indigo obtained differs not only according to the quality of the plant which produces it, but according to the care taken in manufacturing it. However, as indigo in its coloring matter presents but little difference, we conclude that the quality which distinguishes it, depends upon the proportion of foreign matters which are used with it.

CHAPTER II.

COMMERCIAL VARIETIES OF INDIGO.

General Characteristics of Indigo.

Indigo.

Indigo as found in our markets is in pieces sometimes irregular, sometimes in cubic or flat squares, of a blue color, that varies between the violet blue, light blue, cupreous and blackish.

The pieces are dry, firm, easy to be broken; the break is clean, but without brilliance, light, and float commonly upon water. One of the strongest characteristics of indigo is to take by the rubbing of the nail or of a hard substance a metallic brightness and a cupreous reddish tint. The paste is ordinarily fine, homogeneous and light, but sometimes we meet some indigos in which it is rough, or which contain parcels of sand and other foreign substances, or present irregularity of shades, cavities and kind of bubbles which come from an incomplete or too sudden desiccation, or finally it offers some traces of a white matter occasioned by moisture. The consistence of the paste presents also some great differences; there are some dry pastes, hard and compact, others friable and easy to be broken by pressure in large and thin shells. Certain pastes without being soft break easily, and by the simple shaking of the boxes furnish many gargles; this defect seems to come from a badly conducted desiccation, the indigos which present them are called *quartered.*

Indigo in small quantity has no smell, but in large quantity it has a very appreciable one which becomes stronger by being heated or burned, and which may be found in the works of the indigo vat. The taste of indigo is null, but it is in general very absorbing, that is to say if you put the moist tongue upon a piece oi indigo, there results a light adherence and the humidity is almost instantaneously absorbed. This property, which comes from a state of porosity particular to indigo, is often consulted to ascertain the different qualities.

Indigo is a merchandise very difficult to classify, and the different kinds are separated by so delicate shades that it requires much judgment, habit, and practice to distinguish them and place each of them in the rank to which it belongs. In the impossibility to speak of all, we have been careful to choose for subject of our remarks, species well characterized and such that the one described is far from that which precedes and which follows it, of a manner sensible enough to recognize it easily and to perceive at least a difference. To obtain this result we have been obliged to omit the intermediate which connect a kind to another, and those intermediates are four or five times more numerous than even we have described, for example, we speak of 15 varieties of Bengalis

Indigo, and expert judges of this article state as many as 43, that they designate by different names and they know by particular signs.

I. BENGALIS INDIGO.

There exist very numerous differences between the varieties of Bengalis Indigo and the causes of these differences come from the difference in the ground which produced them, the care given to the plant, and extraction of the coloring matter. These indigos are commonly classified as follows:—

Indigo Surfine, light or floating.
Fine Blue.
Blue Violet.
Surfine Violet.
Surfine Purple.
Fine Violet.
Good Violet.
Violet Red.
Ordinary Violet.

Good tender Red.
Good Red.
Fine Cupreous.
Middling Cupreous.
Ordinary Cupreous.
Low Cupreous.

This classification can be increased easily by placing one or two intermediate between each, but then the differences will be difficult to see.

Packing.—This indigo is received in boxes.

1. *Surfine Light Blue.*

Characteristics.—It is in cubic stones sometimes broken, light, friable, of a bright blue color, soft to the touch, break easily, of a clean paste, pure, adhering to the tongue and very spongious.

2. *Fine Blue.*

Characteristics.—Has very near the same characters as the above, same softness, lightness, friability, purity in the paste; the blue color is less bright but well decided.

3. *Blue Violet.*

Characteristics.—It differs from the above in this, that it is less light, friable, and instead of a blue perfectly decided, it reflects a light violet shade.

4. *Surfine Violet.*

Characteristics.—The characters are about the same as the blue violet; the violet shade is more pre-eminent and more easy to remark.

5. *Surfine Purple.*

Characteristics.—Fine paste, light, and reflecting a fine purple color.

6. *Fine Violet.*

Characteristics.—Differs only from the above in this, that its paste is a little less light, and its shade less bright.

7. *Good Violet.*

Characteristics.—This indigo a little less light than the fine violet, is of a thicker paste, and has a shade less pronounced.

8. *Violet Red.*

Characteristics.—Paste heavier and thicker than the good violet; has a violet shade with a reddish reflection.

9. *Ordinary Violet.*

Characteristics.—Thick and heavy paste, violet shade without mixture of other colors.

10. *Good Tender Red.*

Characteristics.—This quality is heavier than the above; the paste has a red reflection, which lessens much of the shades of the fine blues.

11. *Good Red.*

Characteristics.—Thicker paste, and more compact than the above.

12. *Fine Cupreous.*

Characteristics.—Heavier than the good red, thicker and less spongious paste; cupreous shades pretty pure.

13. *Middling Cupreous.*

Characteristics.—Intermediary quality between the fine and ordinary cupreous, thick paste, heavy, difficult to break.

14. *Ordinary Cupreous.*

Characteristics.—This quality is of a red cupreous blue, thick, difficult to break; the paste is not so pure nor so bright.

15. *Low Cupreous.*

Characteristics.—Hard paste, heavy, difficult to break, loaded with impurity, and of a dark cupreous blue.

II. OUDE INDIGO.

This product, improperly called Coromandel indigo, is the product of a province of Industan, inferior in quality to the Bengalis.

General Characteristics.—This indigo is generally quite stony, and is the less easy broken. It owes its solidity to the considerable quantity of lime which enters in its composition.

Packing.—Boxes of different weights.

It is thus classified:—

Violet.
Cupreous.
Ordinary.

1. *Violet.*

Characteristics.—It has generally a thick paste, hard and heavy; its color is a fine violet. By its appearance it resembles certain qualities of Bengalis; it is difficult to distinguish it, except when you come to use it.

2. *Cupreous.*

Characteristics.—Heavier than the above, of a red cupreous color, sometimes blackish, thick paste, hard; containing often sand that you see brighten when you break the indigo, which leaves a considerable deposit.

3. *Ordinary.*

Characteristics.—This kind is in hard cakes, heavy, difficult to break; its paste is sometimes tarnish and earthy, sometimes dirty and slaty blue. It contains much impurities.

III. MANILLA INDIGO.

Characteristics.—This kind is extremely light, in cubic stones, in flat cakes, and often in irregular pieces. It is very mixed.

In the boxes you will find some very fine blue, approaching to the fine Bengalis indigo, with red, cupreous, dry, arid indigos, with tarnish pieces and others called *false* in the trade. It contains a great deal of earth incorporated with the paste.

Packing.—Boxes of different weights.

IV. MADRAS INDIGO.

General Characteristics.—This kind in its superior quality has sometimes the appearance of fine Bengalis; the paste is so light, they differ from them in this, they are less spongious, and when applied on the tongue it takes some time before the humidity is absorbed; they have a cubic form.

Packing.—It is received in boxes.

The divisions adopted in the trade are the following:—

Fine Blue.
Mixed Blue Violet.
Ordinary.

1. *Fine Blue.*

Characteristics.—This quality is in thin paste, light, of a fine light blue and a bright shade; it is the nearest to the Bengalis.

2. *Mixed Blue Violet.*

Characteristics.—It differs from the above in this, that the stones are heavier, some have a blue color, and others have a reddish shade. This kind presents many mixtures and is not homogeneous.

3. *Ordinary.*

Characteristics.—This kind is much mixed, the paste is sometimes ruft, impure, of a pale color; sometimes black, heavy and loaded with sand.

V. JAVA INDIGO.

Characteristics.—This kind is found in the trade in the form of flat cakes, and in trochists it looks as fine as the Bengalis.

Packing.—In boxes.

VI. EGYPTIAN INDIGO.

Its principal varieties are—

Good Violet and Red.
Fine Blue.

1. *Good Violet and Red.*

Characteristics.—Fine and light paste, its color is a mixture of bluish violet, of good violet and good red. Inferior to the Bengalis.

2. *Fine Blue.*

Characteristics.—Lighter than the above, very fine paste, its color is a fine blue a little weak. Sometimes black pieces are found in the boxes.

VII. SENEGAL INDIGO.

This kind is not found enough in the trade to be described and classified. We mention it here only for memory, without giving it any appellation or classification.

AMERICAN INDIGO.

Amongst the American indigos, the best are the Guatemala, Caragua, and Mexican. Some years ago the island of St. Domingo produced a very fine indigo, and some were received from Louisiana, South Carolina, and Brazil, but the culture of this plant has been abandoned in these countries.

These indigos are classified in the following manner: *Flor*, *Sobre*, *Corte.*

VIII. GUATEMALA INDIGO.

General Characteristics.—This indigo is in small pieces, irregular, broken, lighter than water, of a bright blue; its paste is smooth, dense, more easy to break and pierced with an innumerable quantity of very small holes. It is one of the most estimated.

1. *Flor.*

Characteristics.—It has a very fine bright blue color, smooth paste, tender light, absorbs very quickly, humidity is very nearly similar to the fine Bengalis indigo.

2. *Sobre Saliente.*

Characteristics.—Differs from the above in this, it is less light, its paste is firmer and its color less fine. It has sometimes a violaceous shade.

3. *Corte.*

Characteristics.—It has an ordinary Cupreous red color, a thicker, firmer and heavier paste. It resists the fingers in rubbing it.

5

IX. CARAGUA INDIGO.

General Characteristics.—This kind is in irregular pieces, and ranks after the Guatemala; its paste is thick, light, of a soft texture, full of little holes. Its color is sometimes of a fine blue, sometimes violaceous blue.

1. *Flor.*

Characteristics.—Very thick and light paste, of a fine blue color, which is sometimes violaceous; this kind is more full of small holes than the others.

2. *Sobre.*

Characteristics.—The paste is firmer and heavier, and absorbs humidity with more difficulty.

3. *Ordinary Sobre.*

Characteristics.—The paste is heavier and thicker, quality slightly inferior to the above.

4. *Superior Corte.*

Characteristics.—The paste is less thick than the above, pierced inside of small round and numerous holes, it contains some impurities; its color is part violet and part blue.

5. *Good Corte.*

Characteristics.—The paste is heavier, thicker, less fat; it is dry and arid, color violet, violet red and cupreous. More numerous impurities alterate this kind pierced in the inside with round holes.

6. *Ordinary Corte.*

Characteristics.—It is one of the lowest quality; it is dry, hard, ârid, difficult to break, heavy and full of impurities; it presents black and slaty blue pieces.

X. MEXICAN INDIGO.

Characteristics.—This kind is very similar in its aspects to the Guatemala ; its quality resembles the Caragua, then it could be said that it ranks between these two kinds, and is between them an intermediary species.

CHAPTER III.

PHYSICAL PROPERTIES OF INDIGO. COMPOSITION.

Physical Properties.

INDIGO is a solid substance of a violet-blue color, more or less light. It acquires a cupreous color by rubbing it with a hard substance. Generally those that have a tarnish blue color are defective. All commercial indigoes are mixed, and it is difficult to determine their value by their physical properties; those which are defective are designated by the names of *aerated*, *pricked*, *tapid*, *burned*, and *stony*. They are called *aerated*, when the interior break presents a kind of white mixture; *pricked*, when the inside is full of little white specks and little holes; *tapid*, when it presents beds of different shades; *burned*, when in breaking it, it divides itself in pieces more or less black; *stony* or *sandy*, when the inside is full of sand or stones.

Composition.

According to Bergman's experiments, 100 parts of good indigo contain:—

Matters soluble in water	12
" " " alcohol	6
Earthy matters soluble in acetic acid .	22
Oxide of iron	13
Indigotine	43
Lost	4
	100

According to Mr. Chevreul, who made the most complete and accurate analysis, one hundred parts of Guatemala indigo give—

SOLUBLE IN WATER.

Green matter combined with ammonia, White Indigotine, Extractive, Gum,	12

SOLUBLE IN ALCOHOL.

Green matter, Resin, Traces of indigo,	30

SOLUBLE IN HYDROCHLORIC ACID.

Red resin	6
Carbonate of lime	2
Oxide of iron, Alumina,	2

RESIDUE.

Silicic acid	3
Indigotine	45
	100

CHAPTER IV.

CHEMICAL PROPERTIES—INDIGOTINE.

Chemical Properties.

INDIGO is destitute both of taste and smell, and is a neutral body; heated on a platinum foil, it gives a beautiful purple smoke, and if the heat be rapidly increased it fuses, boils, catches fire and burns with a lively flame, gives out much smoke, and leaves a quantity of charcoal difficult to burn.

Insoluble in water, boiled with alcohol, this liquid assumes a blue color, but gradually lets fall a very minute quantity of indigo and becomes colorless. Olive oil and turpentine act precisely the same way. It is insoluble in ether. Neither diluted acids or alkalies attack it.

Chlorine instantly destroys the blue color of indigo and gives it the color of iron-rust. Iodine does not act sensibly when cold, but by the action of heat the color is destroyed. Sulphur and phosphorus do not combine with it.

An alkaline base and dry substance having a strong affinity for oxygen, in contact with indigo deprive it of oxygen; it assumes then a white color and combines with the basis. This process is the one employed in making the calico printer's vat.

Concentrated sulphuric acid dissolves immediately indigo with evolution of heat but without formation of sulphurous acid.

Nitric acid decomposes it rapidly, destroys the color and transforms it into *Indigotic* and *Carbazotic* acids.

Indigotine.

It has long been known that when indigo is heated, it sublimes; the product of the sublimation is pure indigo or *indigotine.*

It sublimes in long flat needles which readily split when bruised into 4-sided prisms; viewed at a particular angle they have the most brilliant and intense copper color, but lying in heaps they have a rich chestnut brown color; besides these needles, indigotine is formed in plates much broader than the needles, and extremely thin, twisted sometimes almost into tubes; viewed obliquely through a microscope they appeared copper colored like needles, but when held perpendicularly they are transparent and of a beautiful blue color.

The vapor is transparent and of a most beautiful reddish color resembling vapors of iodine, but distinguished from it by its shade of red; the sublimation takes place at about 350°. The melting point, its point of volatilization and decomposition are near each other.

Its specific gravity, 1.35.

The crystals heated in open vessels sublimate without leaving a residue, in a close vessel; the vapor is at first reddish violet, but as the heat advances it takes a scarlet color, and before it is entirely decomposed becomes deep scarlet, and then orange color, and a quantity of charcoal is deposited.

Preparation.

The best way to obtain *Blue indigotine* is to have recourse to the calico printer's vat (see vats).

Mr. Walter Crum prepares it the following manner: He employed the covers of two platina crucibles nearly three inches in diameter and of such a form that when placed with their concave side inwards, they were about $\frac{3}{8}$ of an inch distant in the middle; about the centre of the lower lid were placed thinly about ten grains of amorphous indigotine (commercial indigo can be used) then having put on the cover, the flame of a spirit lamp was applied beneath the indigo. In a short time it begins to melt and the vapor to be disengaged, which was known by the hissing noise which accompanied it.

The heat was continued till the noise nearly ceased; the lamp was withdrawn and the apparatus allowed to cool. The sublimed indigo, in removing the crucible, was found planted on its inner surface. In this way Mr. Crum obtained from ten grains of amorphous indigotine,

1.88	grains	of	Sublimated Indigotine.
6.44	"	"	Cinders.
1.68	"	"	Volatile matters.

The following process to prepare sublimated indigotine is due to Mr. E. Kopp, and is the best and cheapest method.

The happy results obtained in the arts of dyeing and calico printing by the use of indigo and madder have induced chemists to make researches for the means of obtaining from the raw materials purer products. All experiments tried have not given products cheap enough to be used in the arts till Mr. E. Kopp has had the happy idea to try for that purpose the superheated steam, and he obtained results which can be of a useful application in industry.

Steam obtained under a considerable pressure without having been submitted to an elevation of temperature posterior to its formation, acts on bodies by the temperature and in the same time as a damp vapor and determines reactions dependent on that dampness. Superheated steam in passing through an apparatus at a temperature varying from 572° to 752° acts as a hot and dry body, without determining reaction that a gas could produce in contact with the same bodies.

Mr. Kopp operates on indigo: he puts it in a metallic cylinder enveloped in another cylinder, in which passes a current of superheated steam of which another portion passes through the indigo. In this way it operates on all parts of the matter which is submitted to the same temperature.

The steam produced by a generation passes through a cast-iron pipe, fixed in a furnace, and before coming in contact with the indigo, through a special capacity divided by a diaphragm pierced with holes and containing a thermometer.

The condensed water takes a little indigotine which can be left to precipitate.

The indigotine so obtained can be used in dyeing and calico printing the same manner as indigo.

Properties.

It is solid, of a purple color, without taste or smell.

Inalterable in the air, insoluble in water, slightly soluble in alcohol, soluble in concentrated sulphuric acid; is decomposed by concentrated or diluted nitric acid.

Hydrochloric acid and alkalies are without action at a cold temperature, but when heated it takes a yellow color due to a little decomposed indigotine.

Chlorine turns it yellow.

Substances void of oxygen and alkalies act on it as on commercial indigo.

Composition.

According to Dumas' analysis sublimed indigo purified by alcohol gives the following composition:—

16 at. Carbon	12.000	or	73.28
5 " Hydrogen	0.625	"	3.82
1 " Nitrogen	1.750	"	10.69
2 " Oxygen	2.000	"	12.21
	16.375		100.00

Its formula is $C^{16}H^{5}NO^{2}$.

CHAPTER V.

ACTION OF ALKALIES AND SUBSTANCES AVID OF OXYGEN ON BLUE INDIGO—WHITE INDIGOTINE.

We have already observed, that when indigo is treated with some reagents capable of absorbing oxygen, it assumes a white or yellowish white color, and becomes soluble in alkalies. Liebig called this white matter *indigogen*, but it is generally known by the name of *desoxygenated indigo* or *white indigo*.

Preparation.

Mr. Chevreul has given the following process to obtain it: in a large stopper vial, introduce 7.75 grains of finely powdered blue indigo with $\frac{1}{2}$ pint of water; make two solutions, one with 28.365 grains of proto-sulphate of iron, and the other with 24.80 grains of pure caustic potash. He introduces these two solutions in the bottle, and fills it with water, so as to have one quart of liquid. He leaves the mixture a few hours in shaking it from time to time. It presents a limpid yellow liquid with a deposit of peroxide of iron. By the contact of the air, this liquid is covered of a violet-blue skim. To explain the phenomena, which pass in this operation, it is sufficient to know that the different substances employed are water, potash, and oxide of iron. Therefore, 9.6875 grains of potash neutralize 8.2305 grains of sulphuric acid, contained in the 28.365 grains of proto-sulphate of iron, to produce 17.9025 grains of sulphate of potash; it is then left 9.6875 grains of free potash, with 7.2075 grains of protoxide of iron, and 7.75 grains of indigo; then the protoxide of iron has a great tendency to combine with oxygen, and takes a part of it to indigo, and the desoxygenated indigotine dissolves in the alkali.

To separate it from its dissolution, decant the clear liquid with a siphon in another vial, in which you introduce without the contact of air, some acetic acid to neutralize the potash; when the deposit is well formed, decant the liquid; filter, and wash the filter with cold boiled water; press the filter between blotting paper, and dry in vacuo.

Properties.

Dry air does not alterate its color, but placed under water, it assumes a deep-blue color, and a copper shade when dried.

It is very little soluble in water, soluble in alcohol and ether; it dissolves in alkalies without neutralizing them.

Nitric acid turns it blue.

In solution in an alkali and brought in contact with the air, the oxygen gas is absorbed and blue indigotine regenerated and precipitated on form of a blue powder; in this operation it absorbs 11.38 per cent. of oxygen.

Dissolved in alkalies it unites by double decomposition to alumina, oxides of iron, tin, and lead. These compounds are insoluble and turn blue in the air; salts of copper turn blue the solution of white indigo in oxidizing it.

Composition.

If we consider the atomic weight of indigo 16.375, and admit that it contains two atoms of oxygen, and that white indigotine is entirely destitute of oxygen, its atomic weight will be 13.625. Now 404 : 47 (the increased weight of the white indigo) :: 13.625 : 1.58. It appears from this, that the quantity of oxygen absorbed by the atom of white indigo is only 1.58 or 0.42 less than 2 atoms.

The experiments of Berzelius do not agree with those of Liebig; he finds that when a solution of indigogen was mixed with that of copper the whole is converted into blue indigo by absorbing oxygen from the salt and that two parts of indigo had absorbed 4.6 of oxygen. Now 4.6 is nearly $\frac{1}{5}$ of the whole oxygen contained in 160 parts of indigotine, so that regenerated indigo, according to his experiment, differs from white indigotine by only $\frac{2}{3}$ of an atom of oxygen; but Liebig's experiment is better entitled to confidence.

Mr. Dumas has resumed the subject; he analyzed white indigotine and found the following composition:—

16 at. Carbon. . . .	12	or per cent.	72.73
6 " Hydrogen . . .	0.75	" " "	4.54
1 " Nitrogen	1.75	" " "	10.61
2 " Oxygen.	2.00	" " "	12.12
	16.50		100.00

According to this analysis it is an *hydrate of indigo* or indigo united to one atom of hydrogen.

Dumas has entered into no details, but he has full confidence in his results; chemists, however, cannot adopt those conclusions till details are furnished which enable them to judge the accuracy of the analysis.

CHAPTER VI.

ACTION OF SULPHURIC ACID ON INDIGO.

When you heat one part of indigo with twelve parts of sulphuric acid, the color turns yellow in some parts; it passes green, and at last it takes a very dark-blue color. Berzelius has ascertained three products in this compound, called by Chevreul *sulpho-indigotic acid*, *hypo-sulpho-indigotic acid*, and *sulpho-phenicic acid*. Walter Crum has called the blue substance *cerulin*.

The mixture of the blue substance with sulphuric acid is a semi-fluid which requires much water to dissolve it. The cerulin is precipitated from it by any salt of potash, and the precipitate is a combination of cerulin and sulphate of potash. Crum distinguishes it by the name of *ceruleo-sulphate of potash;* this salt is soluble in pure water.

Salts of soda form also precipitate in the solution of cerulin with sulphuric acid, and these are likewise insoluble in solutions of potash and soda, though soluble to a certain extent in pure water. When heated, these *ceruleo-sulphates* dissolve even in solutions of their salts. On cooling the solutions, part falls down in blackish grains, a portion, however, remaining in solution. The soda compound is more soluble than that of potash.

The salts of ammonia form precipitate in the sulphuric dissolution of cerulin.

The precipitate dissolves in hot solutions of salts of ammonia, and separates by cooling. The precipitate seems to be a combination of cerulin with sulphate of ammonia; hot water dissolves it abundantly. It is soluble between forty and fifty times its weight of cold water. Similar compounds may be formed with baryta, strontia, lime, and with most of the bases.

Ceruleo-sulphate of potash has so deep a *blue* color, that when wetted with water, it appears *black;* dry, it has a shining copper-red color, by transmitted light it is blue; it attracts water from the air. Cold water dissolves $\frac{1}{140}$ of its weight of this substance, and forms a solution so deeply colored, that when diluted with twenty times its weight of water, in a vial one inch diameter, it may be just seen transparent.

Water in a wine-glass containing $\frac{1}{500000}$ths of its weight is distinctly blue; chloride of tin turns it yellow in deoxidizing the indigo; it becomes blue again by addition of any salt capable of imparting oxygen to it.

When indigo is dissolved in sulphuric acid considerable heat is produced, but there is no evolution of sulphurous acid. The solution is at first yellow; if it be dropped into water it instantly becomes blue and the indigo precipitates unaltered; if the solution be kept undiluted for 24 hours the indigo is transformed into cerulin.

Walter Crum has drawn from his analysis the following composition for cerulin:—

1 at. of Indigo	15.625	=	77.639
4 " " Water	4.500	=	22.361
	20.125	=	100.000

Berzelius has taken a different view of the action of sulphuric acid on indigo.

According to him, when indigo is dissolved in sulphuric acid, a combination takes place between them and two new acids are formed, the *hypo-sulpho-indigotic acid* and the *sulpho-indigotic acid.* The stronger the sulphuric acid employed, the greater is the quantity of the first of these acids formed and the smaller the second. English sulphuric acid gives more sulpho-indigotic acid than the Nordhausen acid.

Mr. Dumas has lately examined this solution of indigo. He finds it to possess the property of an acid, and has given it the name of *sulph-indilic acid,* and assures it is a compound of—

1 at. Indigo	16.375	=	62.085
2 " Sulphuric acid	10.000	=	37.915
	26.375	=	100.000

The *Sulphindilate of Potash* is composed of—

2 at. Sulphuric acid .	10.000		30.888
1 " Indigo . . .	16.375	=	50.580
1 " Potash . . .	6.000	=	18.532
	32.375	=	100.000

The purple matter which precipitates when indigo is dissolved in sulphuric acid is, according to Dumas, a compound of—

2 Indigo	=	32.75	=	76.609
2 Sulphuric acid	=	10.00	=	23.391
		42.75		100.000

It possesses acid properties, and has been called by Dumas *Sulpho-purpuric acid*, which forms with potash a purple salt composed of—

1 Sulpho-purpuric acid.
1 Potash.

These two acids of Dumas are identical with the two of Berzelius.

Sulphuret hydrogen gas reduces the indigo in those acids, hence the reason why the liquid has a yellow color when the sulpho or hypo-sulpho-indigotate of lead is reduced by that gas.

According to Mitscherlich, when these acids are saturated with a base, the sulphuric acid only combines with it, and the indigo (*cerulin* of CRUM) acts in a way similar to the water of crystallization of simple salts.

Crum in his researches discovered that if the action of sulphuric acid be stopped to a certain point, a new substance is formed, possessing singular properties; it is formed at the instant that indigo changes from yellow to blue by the action of sulphuric acid. Crum called the substance *phenicin,* and Berzelius gives it the name of *purple of indigo.*

Crum obtained it in the following manner: he mixed one part of purified indigo with seven or eight parts of concentrated sulphuric acid in a stoppered bottle, and leaves the mixture to itself till it becomes of a bottle-green color. He mixed it with a large quantity of distilled water, and threw it upon a filter; by continuing to wash the precipitate with distilled water, the liquid, at first colorless, becomes more and more blue, and after some time the indigo, which had been changed, passes through. The colorless washings were thrown away. The blue liquid which contains the phenicin did not differ in appearance from a solution of cerulin.

By addition of chloride of potassium, the phenicin is precipitated on form of a most beautiful reddish purple color, similar to the vapor of indigo. It was thrown on a filter, and washed with distilled water, till the liquid that passes through began to form a reddish precipitate with nitrate of silver; it was then dried.

Dried, it has a brownish-black color; it dissolves in water and alcohol; its solutions are blue. Alkaline substances precipitate it from those solutions with its original purple color.

Acids have no effect in preventing the precipitation of phenicin by salts, and the precipitates once formed are not redissolved in the same liquid by the assistance of heat.

Crum has analyzed it and found it composed of—

1 Indigo,	15.625	=	87.412
2 Water,	2.250	=	12.588
	17.875	=	100.000

He has given the following method to prepare it in greater quantity, though not so pure.

1. Mix together, in a vial, one part indigo in powder, with ten parts of concentrated sulphuric acid.

2. Agitate it for some time, till the blue color which the indigo loses at first, is completely restored. In summer it requires three hours, and at 100° it is effected in about twenty minutes; at 45° ten or twelve hours are necessary.

3. Pour this mixture in a large quantity of distilled water, and filter.

4. Take the precipitate of the filter, and wash it well with distilled water containing as much salt of ammonia, as will prevent the substance to dissolve in.

5. Collect it again on a filter.

6. Dissolve the precipitate anew in a large quantity of distilled water; heat the dissolution

to drive off the air, and let stand two or three days in a tall vessel.

7. Draw off the clear, and wash the remainder with more distilled water.

8. To the solution add any alkaline salt, till all the phenicin is precipitated, and wash with distilled water till the liquid refuses to pass through.

Phenicin dissolves in liquid ammonia without injury; fixed alkalies destroy it, though not readily. Chloride of tin precipitates the solution, but gradually redissolves the precipitate in forming a yellow solution. It dissolves readily in concentrated sulphuric acid, and if poured immediately in water, it is precipitated again. If the action of sulphuric acid is prolonged, it is converted into ceruline.

Such are the properties of ceruline as described by Crum, to whom we are indebted for our first knowledge of it.

CHAPTER VII.

SULPHO AND HYPO-SULPHO-INDIGOTIC ACIDS—SULPHATE OF INDIGO—SOLUBLE INDIGO—CARMINE OF INDIGO.

THE compounds called *sulpho* and *hypo-sulpho-indigotic* acids were examined first by Berzelius.

Preparation.

To obtain them, dissolve indigo in fuming sulphuric acid; dilute the solution with 30 to 50 per cent. of water, and pass the liquid through a filter; pieces of flannel perfectly clean are to be put into the solution; the flannel gradually combines with the blue-colored acids, and assumes a deep blue color;

take it out and introduce successively other pieces of flannel, till all the coloring matter has been successively absorbed. If, instead of indigo, you have used indigotine, nothing will remain but acidulated water. Wash the pieces of flannel in pure water, till that liquid ceases to acquire an acid taste. Digest them in water, in which some carbonate of ammonia has been dissolved. These acids separate from the flannel, and combine with ammonia and the liquid assumes a deep-blue color; the liquid is decanted, and the flannel well washed in pure water. If the flannel continue still to be colored, digest it a second and even a third time with water containing carbonate of ammonia in solution. Evaporate the ammoniacal liquid to dryness at the temperature of 140°, and pour on the residue alcohol at 0.833 of specific gravity; it dissolves the *hypo-sulpho-indigotate of ammonia*, and leaves the *sulpho-indigotate* undissolved. To obtain the *sulpho-indigotic* acid, dissolve the sulpho-indigotate of ammonia in water, and precipitate by acetate of lead. The sulpho-indigotate of lead being insoluble falls to the bottom; collect it on a filter; wash it well; mix the salt of lead with water, and decompose it by a current of sulphuretted hydrogen gas; the liquid is yellow, and turns blue by the contact of the air; filter; wash the precipitate, and evaporate at a temperature not exceeding 122°; it leaves a solid residue of a dark-blue color, which is the *sulpho-indigotic acid.*

Properties.

This acid attracts the humidity of the air; is soluble in water and alcohol; those solutions have a deep blue color; it has a peculiar, but agreeable odor, similar to that observed when reduced indigo becomes blue by exposure to the air. Its taste is acid and astringent.

Preparation of Hypo-sulpho-Indigotic Acid.

This acid is obtained by mixing the above alcoholic solution with an alcoholic solution of acetate of lead; a blue salt of lead is precipitated, which being treated the same way as the sulpho-indigotate of lead, gives the pure acid.

Properties.

When hypo-sulpho-indigotic acid is evaporated, it dries quite well at the edges, but in the middle the mass remains moist, and attracts some moisture from the air; its taste is acid, and its other properties are similar to those of the sulpho-indigotic acid.

Those acids are decomposed by heat into sulphurous acid, sulphite of ammonia, much water, and traces of volatile oil; the sulphite of ammonia becomes blue when dissolved, probably in consequence of a little indigo carried with it.

When zinc or iron are introduced into these acids, the metal becomes oxidized at the expense of indigo, and if we have employed an excess of acid, we obtain a yellowish colored liquid, containing a salt of zinc or iron combined with the reduced indigo and soluble, which becomes instantly blue when in contact with oxygen or air. Sulphuretted hydrogen gas has the property to reduce the indigo contained in these two acids. Chloride of tin, by action of heat, destroys also the blue of those acids.

Sulpho-Indigotate of Potash.

It may be obtained by digesting the above blue flannel in water containing carbonate of potash; the solution is evaporated to dryness, and the residue treated by alcohol, which dissolves the *sulpho-indigotate of potash*, and leaves the excess of carbonate of potash. If we saturate sulpho-indigotic acid with carbonate of potash, and add a small excess of this last salt, the liquid is converted into a kind of jelly.

This salt is readily soluble in boiling water, and precipitates partially in flocks while cooling. Cold water dissolves $\frac{1}{440}$th of its weight, assuming so deep a blue color as to become opaque. When the solution is evaporated, the salt remains with a cupreous lustre.

This salt has received various names: Bergman called it *precipitated indigo* (*indigcarmin*). In France it is called *soluble indigo.*

Sulpho-Indigotate of Soda and Ammonia.

They are analogous to the preceding, but they are precipitated less completely. They are prepared the same. The ammoniacal salt is more soluble than those of potash or soda.

Sulpho-Indigotate of Baryta.

When the sulpho-indigotate of potash is mixed with chloride of Barium, the salt precipitates in flocks of a blue color. It is not completely insoluble in water; it dissolves in boiling water, but is precipitated by cooling. This salt is not precipitated by a small quantity of sulphuric acid.

Sulpho-Indigotate of Lime.

It is obtained by diluting the sulpho-indigotic acid with forty or fifty times its weight of water, and saturating it with white marble in powder, till it becomes neutral; filter, and wash the sulphate of lime on the filter till it becomes red. The solution is evaporated, and mixed with alcohol; it produces a precipitate which is collected on a filter, and washed with weak alcohol. Dry, it has a deep-blue color.

Sulpho-Indigotate of Alumina.

This salt is soluble in water, and may be dried like the above salts.

Sulpho-Indigotate of Lead.

We have seen how this salt can be obtained; it is in flocks, and slightly soluble in water:

when dried, its color is almost black; its taste is astringent, but not in the least sweet.

Sulphate of Indigo of the Dyers.

Having examined the action of sulphuric acid on indigo, we have to give the preparations used in the shops, and as these proportions and preparations are different from those indicated by Mr. Capron de Dole, we shall give them here.

Take one pound of finely powdered indigo; dilute it with four pounds of fuming sulphuric acid; leave the mixture in contact for twelve hours in shaking it from time to time; place the vases in a water bath, and leave it in for twelve hours in shaking; take out and leave to cool. To keep this dissolution, dilute it with four or five times its volume of water.

Distillate of Indigo.

The solution of indigo being obtained, dilute it with 25 gallons of water, and when dissolved introduce into it perfectly clean wool, and leave it 6 hours, or all night.

Wash the wool with water and boil it with water containing four ounces of carbonate of soda.

Carmine of Indigo.

Prepare a solution of indigo with one pound of indigo, and five pounds of acid, in operating as above. Dilute the solution with fifteen times its volume of water, saturate the acids by carbonate of soda, in adding it slowly and by small portions at a time and stop when the saturation is complete; filter and wash the precipitate first with water containing sulphate of soda in solution, and at last with pure water, till this water passes blue. This blue is in paste and has a cupreous reflection.

In the following chapters we shall enter into some details on the preparation of the blues of indigo, which is given entirely from the work of Mr. Capron de Dole.

CHAPTER VIII.

DESCRIPTION OF THE NECESSARY TOOLS FOR A LARGE FABRICATION OF CARMINE OF INDIGO.

1. *A lead pot* widened in its superior part, of a capacity of 6½ to 8 gallons. It is used to make the sulphuric dissolution of indigo.

2. *A Mill* to grind the indigo as fine as possible.

3. *Cylinders* to grind the paste of blues after they have been kneaded once.

4. *A pestle* to grind the pastes from which you make the blues in balls.

5. *A glass or wood rod* lined with lead, to stir the sulphuric dissolution of indigo; a common wood stick will blacken the dissolution on account of the sulphuric acid which acts on the wood.

6. *Three small tubs*, the height of an ordinary barrel, of a capacity of about 62 to 78 gallons. Each one has a large cock, and is fixed on threepods about 12 inches in height.

7. *Twelve wooden frames* about 27 inches square with little hooks to fix the filters.

8. *Twenty-four filters* in white felt of about $1.60 to $1.80 the yard, of the size and wideness of the interior of the frame, on which they are fixed with three hooks on each size.

After each operation those filters must be well washed and substituted by some other, that is the reason why 24 are needed and only 12 frames. Before using them for the first time they must be well washed with boiling soap water, and every time that you want to use them they must be perfectly wet.

The frames with the filters are fixed one near the other on two pieces of wood about 9 yards long, which are equally fixed parallely and horizontally on trestles about 2½ feet in height so to put one pail under.

9. *Twenty pails* at least.

10. *Four other filters* of a stronger cloth than the above, to put the carmine in press.

11. *A press* by weight fixed on a table a little bented so that the water which goes out from the carmine could run easily.

To fix this press you have a table having the form of a long square well fixed on the floor by its trestles, of which one must be necessarily larger than the table, on the edge of the trestle which goes beyond the wideness of the table, you adapt with solidity a large piece of wood about 1 foot in height and half of which at least in form of tenon with different holes; afterwards you have another piece of wood about 2 yards long, one end of which is provided with a mortise crossed by a hole to fix it with an iron piece on the piece of wood elevated at the edge of the trestle of the table. Then you have only to put a weight at the end of the large piece of wood and you have a very economical press.

12. *Several vessels of red copper* to draw the blue or rather the liquid contained in the little

tubs, after the saturation, and put it on the filters.

To be useful, those vessels must be widened at the superior part, and of a capacity of about four to five gallons, and provided with a beak.

13. *Two little shovels* of red copper, having the form of a skimmer, to take out the paste or the carmine from the filters.

14. *A large pot* to wash the carmine.

15. 150 to 200 *small boards* well polished, of 1 yard long and 1 foot wide, to mould the different pastes of carmine.

16. *A table*, having the form of a long square with a press on the top to mould the blues in stones, pastils, etc. This table is fixed on three trestles about four feet high; the middle one must pass off every side of the table, and must be surmounted with two pieces of wood, one of which is about 1½ foot high, and provided at about 4 inches high with a tenon with several holes, and the other of about 8 inches high, provided with a tenon at 4 inches high, but without holes.

On those two pieces of wood placed at each end of the trestle, is fixed by mortise at every end, another piece of wood, in the middle of which you make another mortise of 4 to 5 inches long, on 2 to 3 inches wide to place the beak to mould. This large piece of wood is consequently found placed across the table.

After the piece of wood of which the tenon which surmounts it, is provided with different holes, you adapt to it, by an iron pin and by a mortise traversed by a hole, another large piece of wood 2 yards long, and you have a press to mould.

17. *A beak* to mould surmounted with a skin bag about 12 inches high.

The skin bag must be sewed with a double stitching, and the beak to mould is put on the piece of wood across the mould, and in the mortise.

18. *Some Sheet iron horns* to mould the buttons, pastils, etc. The end of the horn is the same size that the goods, and they are provided with little pieces of wood called *runners*, to push the paste put in the horn, and make it

pass it by the little hole, which give it the form and size desired.

19. *A kneader* to knead the different pastes.

20. *A round knife* to cut the pastes when they are moulded on the little boards.

21. *Several little pots* of red copper to draw the water necessary to the saturation.

22. *A skin bag* 1 yard long, on 8 inches wide, to pass the dried goods to the prussian blue. We call pass to the blues, the blues to which it is necessary to give the celestian blue color.

23. *A sheet iron drum* provided with a door, fixed on two trestles, and put in motion by a crank, to pass the blues in balls, stones, pastils, etc., to the blue of indigo.

24. *A woollen bag* to polish; same size as the above.

25. *An oven* in which are disposed places to receive the small boards, on which you have moulded the blues so as to dry them.

CHAPTER IX.

SULPHURIC DISSOLUTION OF INDIGO—SATURATION—CARMINES OF INDIGO.

THE quantities of acid and indigo indicated in this chapter, are those employed in almost every manufacture.

To manufacture good carmine, put in the lead vessel 22¾ lbs. of Saxony or Nordhausen sulphuric acid.[1] Throw slowly in this acid, and by small portions, 6¼ pounds of indigo powdered as fine as possible, being careful to stir with the glass rod all the time that you throw the indigo, so as to prevent it from adhering to the bottom of the pot, which would

[1] When you want to employ ordinary acid, you use 66 ounces for 16 ounces of indigo.

9

burn it, and prevent its making the mixture. This operation is called the making of the *sulphuric dissolution of indigo.*

When you have thrown all the indigo in the acid, cover the pot with a wooden covercle, and surround it with woollen cloth to concentrate the heat; put it in a water bath for about twelve to fifteen hours. The lead pot must not touch the bottom of the water bath, because the indigo will burn. The water bath must be always full of boiling water.

The mixture of indigo and acid is shaken from time to time with the glass rod.

It can occur that the mixture raises up; then you must diminish the heat and stir at the top only.

After twelve or fifteen hours of staying in the water bath (the time depends on the degree of the acid and heat of the bath), try if the dissolution is completed, *i. e.*, if the indigo is all dissolved; for that you have a felt filter fixed on a frame; dilute some of the dissolution, and filter it. If nothing stays on the filter, the dissolution is complete; in the contrary case put back in the water bath, and heat it till perfect, and that the solution can pass through the filter.[1]

When the dissolution is complete, they put it in a jar, and dilute it with four or five times its weight of water, and they put it by equal portions in the little tubs used for the saturation.

This operation done, fill a caldron with crystals of soda and water; heat in order to dissolve the soda; when it is dissolved and the alkaline solution is boiling, pour it by one or two quarts a time and every ten or fifteen minutes on every part of dissolution of indigo which is in the small tubs, and continue till complete saturation.

You are sure all the liquor is saturated when it does not redden litmus paper.

The saturation being achieved, draw in the vessels of red copper the liquid contained in the

[1] Manufacturers generally, to ascertain if the dissolution is perfect, put some in a glass of water; if it divides well and does not precipitate, they consider it good.

little tubs, and put it on filters; it stays on the filters a paste of a cupreous color that you collect with the shovels; this paste is called *Carmine of indigo.* This carmine is not pure, it contains foreign matters; to have it perfectly fine put it in a jar with three pails of water, shake about half an hour with a wooden spatula.

The mixture being well done, throw it on filters, and when all the water has well dropped, divide this carmine into four parts about equal, and put every part into filters with a handful of Epsom salt.

Put those filters under the press two by two parallelly; the two above are separated from the two below by small boards.

On the two above packages place a piece of board, on which is the arm of the press that you load first with at least 40 lbs. and at last with 60 lbs.

The weight of 60 lbs. must not be put till the carmine is a little purified. Leave this carmine about two days under the press (the time varies according to the number of lbs. you will reduce it, and the quantity of goods you will manufacture), and every twelve hours turn it over with a little shovel, being careful to cover it with some Epsom salt. At last for the manufacture of the blues used to azure, the carmine of 6¼ lbs. of indigo must be reduced at, viz:—

1st. *At* 17½ *lbs. for the blues known in the trade as* EXTRA FINE.

2d. *At* 19½ *lbs. for those called* SURFINE.

3d. *At* 24¾ *lbs. for those called* FINE.

For those known by the numbers 1, 2, 3, 4, 5, the carmine is not washed nor purified under the press, it is only left to purify on the filters, so that the carmine is put in the following quantities:—

Number 1, . . .	at 35 pounds.
" 2, . . .	" 41¼ "
" 3, . . .	" 49½ "
" 4, . . .	" 66 "

For the number five it is not left to purify.

When you wish carmine of indigo, even at 40 cents per pound, leave it to purify only a short time on the filters; afterwards dilute the carmine with saline water which comes from sulphate of soda, that you dissolve in a barrel (this saline water must not mark more than 9 to 10° Baumé to the areometer).

To sell liquid carmine even at 40 cents per pound, many manufacturers dilute them with pure water, but in sending them to their customers in little barrels, the carmine precipitates and separates from the water; it is not the same with this saline water, the acid contained in the salt keeps the carmine in suspension, and does not leave it to precipitate.

CHAPTER X.

CUPREOUS OR BRONZE BLUES—BLUES IN STONES —PASTILS AND STREAKED PASTILS.

THESE different blues that the manufacturers, to give them more value, called *old blues*, *new blues*, *cupreous blues*, *bronze blues*, and other names useless to enumerate, are prepared all the same manner, the only difference is in the more or less gum or fecula employed, the form and polishing.

To prepare them in the different qualities, they use the carmine in the proportions indicated in the above chapter, and the gum or fecula in the following:—

Extra Fine Blues.

Carmine reduced at $17\frac{1}{2}$ lbs.	17 lbs.	8 ounces.
Powdered gum . . .	13 "	8 "
Fecula	5 "	2 "
	36 "	2 "

Surfine Blues.

Carmine reduced at . .	19 lbs.	8 ounces.
Gum	16 "	8 "
Fecula	7 "	4 "
	43 "	4 "

Fine Blue.

Carmine reduced at . .	24 lbs.	12 ounces.
Gum	20 "	10 "
Fecula	8 "	4 "
	53 "	10 "

Blue No. 1.

Carmine purified at . .	35 lbs.	1	ounce.
Gum	26 "	13	ounces.
Fecula	12 "	6	"
	74 "	4	"

Blue No. 2.

Carmine purified at .	41 lbs.	4	ounces.
Gum	33 "	0	"
Fecula	14 "	7	"
	88 "	11	"

Blue No. 3.

Carmine purified at . .	49 lbs.	8	ounces.
Gum	39 "	3	"
Fecula	20 "	10	"
	109 "	5	"

Blue No. 4.

Carmine purified at . .	66 lbs.	0 ounces.
Gum	45 "	6 "
Fecula	30 "	15 "
	142 "	5 "

Blue No. 5.

Carmine purified at about	92 lbs.	13 ounces.
Gum	72 "	3 "
Fecula	82 "	8 "
	247 "	8 "

Those last blues must never be used; they dirty cloth instead of azuring them.

When you wish to manufacture them, put the carmine with the gum and fecula in the kneader, and knead with the hands, so to have a perfect mixture; afterwards pass the paste once or twice through the cylinders, and when well prepared you cannot distinguish in it fecula or gum; mould it into the desired form in the beak, and with the press on the small boards; then cut it the size you want, and dry in the oven.

To use the press, it needs two men, one to press and the other to draw the small boards that you put on the small pieces of wood provided with small wheels.

You must not dry too quick, and not leave current of air in the oven; otherwise the goods will break.

These goods have a dirty blue color; then to give them the aspect of copper or bronze, you put in the sheet iron drum powdered indigo with about one pound of those goods; move the drum for 15 minutes, and when well covered with indigo dust, put them in the woollen bag; that you shake well for 15 minutes. After these operations those goods have a fine cupreous blue color, and they are put in paper of different shapes to be sold.

CHAPTER XI.

CELESTIAN BLUES, ALSO CALLED NEW BLUES AND SOLUBLE BLUES.

THREE qualities of celestian blues are only manufactured on forms of buttons, pastils, etc.; the only difference which exists between those and those spoken of in the other chapter is, that to the carmine, gum, and fecula they add sulphate of potash or soda reduced in very fine powder. Sulphate of soda is better than sulphate of potash on account of its lower price.

To manufacture celestian or soluble blues, take the following proportions:—

Extra Fine Blues.

Carmine reduced at . .	17 lbs.	8 ounces.
Gum	6 "	3 "
Fecula	5 "	2 "
Sulphate of soda . .	12 "	6 "
	41 "	3 "

Surfine Blues.

Carmine	19 lbs.	9 ounces.
Gum	8 "	4 "
Fecula	6 "	3 "
Sulphate of soda . .	15 "	7 "
	49 "	7 "

Fine Blues.

Carmine	24 lbs.	12 ounces.
Gum	11 "	5 "
Fecula	7 "	3 "
Sulphate of soda . .	18 "	9 "
	61 "	13 "

You knead all those matters, and pass them through the cylinders the same as for the blues in stone, and when the paste is well rubbed, mould it with the horns on the small boards, the same as confectioners prepare lozenges. Dry in the oven.

Those pastils, buttons, etc., when dried, have, like the blue in stone, a dirty blue color; to give them a celestian-blue color, put them by two pounds at a time in the skin bag with powdered Prussian blue; shake this bag for about ten minutes; then take them out, and they are ready for the market.

CHAPTER XII.

BELARD OR SAXONY BLUES.

THOSE blues are found in the trade in form of little smooth pastils, and are prepared in the same manner as the blue in stones, etc., except that instead of using potatoes fecula they employ common starch.

To manufacture them employ the following quantities:—

Extra Fine Blues.

Carmine reduced at . .	17 lbs.	8 ounces.	
Gum	12 "	6	"
Starch	6 "	3	"
	36 "	1	"

Surfine Blues.

Carmine reduced at . .	19 lbs.	9 ounces.
Gum	14 "	7 "
Starch	9 "	4 "
	43 "	4 "

Fine Blues.

Carmine reduced at . .	24 lbs.	12 ounces.
Gum	16 "	8 "
Starch	12 "	6 "
	53 "	10

Blue No. 1.

Carmine reduced at .	35 lbs.	1 ounce.
Gum	22 "	11 ounces.
Starch	16 "	8 "
	74 "	4 "

Blue No. 2.

Carmine reduced at .	41 lbs.	4 ounces.
Gum	28 "	14 "
Starch	20 "	10 "
	90 "	12 "

Blue No. 3.

Carmine reduced at .	49 lbs.	8 ounces.
Gum	30 "	15 "
Starch	28 "	14 "
	109 "	5 "

The numbers 4 and 5 are never manufactured.

CHAPTER XIII.

BLUES IN BALLS.

THESE blues are prepared differently from the above; in their composition, gum, fecula, starch or sulphate of potash does not enter, but fine Spanish chalk.

This chalk, before using it, must be put in digestion in water and shake from time to time.

It is used in the following proportions:—

Extra Fine Balls.

24 lbs. 12 ounces.

Surfine Balls.

31 pounds.

Fine Balls.

37 lbs. 2 ounces.

Balls No. 1.

45 lbs. 6 ounces.

Balls No. 2.

55 lbs. 11 ounces.

Balls No. 3.

72 lbs. 3 ounces.

Balls No. 4.

88 lbs. 11 ounces.

Balls No. 5.

103 lbs. 2 ounces.

Thus to manufacture blue balls, mix according to the quality you will make, to the sulphuric dissolution of indigo, the chalk in the proportions indicated above, stir well with a shovel, and when done, the paste which has a gray dirty blue color, is very hard and compact; then rub it well with the hand; this paste becomes soft; mould it in small balls and dry it in the oven.

To make those balls of equal weight and size, the manufacturer spreads the paste on a table and cuts it in equal pieces by the way of a little sheet-iron frame equally divided.

Those balls being dried, have, like the blue in stones, etc., a dirty blue color, then to give them the appearance of cupreous blue, they are put 2 lbs. by 2 lbs. in the sheet-iron drum with very fine powdered indigo; shake this drum for ten minutes, take out and put in the woollen bag, that you shake for about 15 minutes; at last they are submitted when dried to the same operations as the blues in pastils, etc. etc.

CHAPTER XIV.

LIQUID BLUES—FAMILY BLUES.

THE liquid blues are nothing else than the sulphuric dissolution of indigo, saturated or not, diluted with pure or saline water from the sulphate of soda in more or less quantity, according to the price they are to be sold at.

When a family wants to make its own blue to azure, put 8½ ounces of ordinary sulphuric acid in an earthen jar of two quarts capacity; throw slowly, and by small portions in this acid, 4 ounces of indigo in very fine powder; shut the pot, and put it in a water bath for about ½ a day, in shaking from time to time; when the indigo is well dissolved, put in a pail 2 pounds of crystals of soda; that you dissolve in water; then pour that water by small portions in the sulphuric dissolution, and then dilute with water according to the shade you wish to have, and keep the whole in vases. Then for about sixty or eighty cents you can make about 20 quarts of liquid blue that you have to dilute with much water before using it.

CHAPTER XV.

ACTION OF NITRIC ACID ON INDIGO.

THE action of nitric acid on indigo produces *Isatin*, $C^{16}H^{5}NO^{4}$, remarkable for the numerous substances derived from it. A liquid paste is made with two pounds of indigo, and water which is carefully heated in a porcelain capsule, nitric acid being gradually introduced with constant stirring until 20 or 25 ounces of acid are added; the indigo has then disappeared, and the liquid, which is more or less brown colored, contains the isatin mixed with several other substances not examined yet. The liquid being diluted with a large quantity of water is heated to boiling, and the boiling liquid rapidly filtered; when the isatin is deposited, on cooling, in reddish crystals. The deposit remaining is heated with the mother liquid which has deposited the first crystallization of isatin which furnishes an additional quantity, and this process is repeated until no more isatin is deposited.

Isatin may also be obtained by heating indigo with a mixture of bichromate of potash and sulphuric acid; dissolve in 20 or 30 parts of water.

Isatin is slightly soluble in cold water, but largly so in boiling water, and still more freely in boiling alcohol. Its solution does not act upon Litmus. Heated, it first melts, gives off vapors of unaltered isatin; the greater portion of the substance being nevertheless decomposed, and leaving a copious carbonaceous residue. Concentrated cold nitric acid readily dissolves isatin, forming a brownish red liquid which deposits unaltered isatin, while if the liquid be boiled, lively reaction ensues, and oxalic acid is formed.

Isatin is easily acted on by chlorine, and yields products derived by substitution. The isatin must be diluted with water, and a current of chlorine gas passed through, when *mono-chlorinated isatin*, $C^{16}H^{4}ClNO^{4}$, is first formed, while if the action of chlorine be prolonged *bi-chlorinated isatin*, $C^{16}H^{3}Cl^{2}NO^{4}$, is produced; the same compounds being obtained by causing chlorine to act on indigo.

When a concentrated solution of potash is poured over isatin, there results first, a violet colored liquid, which, by boiling, and after being diluted with water, is converted into a yellowish solution depositing crystals on evaporation. Here isatin has seized upon the elements of 1 equiv. of water, and been converted into a new acid called *isatic acid*. The formula of *isatate of potash* being $KO,C^{16}H^{6}NO^{5}$.

By submitting isatin to action of reducing agents, it is changed into *Isathyd*, $C^{16}H^{6}NO^{4}$, by a reaction exactly similar to that which converts blue into white indigo—Sulphydrate of ammonia being poured into a hot alcoholic solution of isatin and the mixture being allowed to rest for a few days, sulphur is deposited at the same time with laminated crystals of isathyd which are colorless or slightly grayish. They are insoluble in water, but slightly soluble in boiling alcohol, from which they are deposited in cooling, and they are decomposed by heat. By treating mono and bi-chlorinated isatin in the same manner, there result *mono-chlorinated Isathyd*, $C^{16}H^{5}ClNO^{4}$, and *bi-chlorinated Isathyd*, $C^{16}H^{4}Cl^{2}NO^{4}$.

If sulphuric acid gas be substituted for sulphydrate of ammonia, the isatin is not satisfied with one equivalent of hydrogen, but also exchanges 2 equivalents of oxygen for 2 equivalents of sulphur, and furnishes a new substance, the *Bi-sulphisathyd*, $C^{16}H^{6}NO^{2}S^{2}$, which, when heated with an alcoholic solution of potash, forms a red liquid depositing colorless crystals of *sulphisathyd*, $C^{16}H^{6}NO^{3}S$.

If, on the contrary, the bi-sulphisathyd be heated with a highly concentrated solution of potash, the two equivalents of sulphur are removed and a rose-colored liquid is obtained holding a rose-colored substance in solution, of the same elementary composition with white indigo, and which has received the name of *Indin.*

CHAPTER XVI.

DYEING WITH INDIGO VAT.

Preparation of Vats.

THE preparation for dyeing blue is not done in kettles like the other colors, but in large wooden vessels called *vats* which are established in a place rendered proper to keep the heat. In many shops they now prepare a copper vat fixed in the ground, and which is buried about 24 inches from the bottom; at this height they made a furnace without grate, where they pass a steam-pipe to heat round the kettle so to keep the bath always at 86 or 122°.

We distinguish under the name of dyeing

bath three kinds of vats: 1°, the vat with lime and sulphate of iron; 2°, the blue stone vat; 3°, and the woad vat.

The vat with green vitriol can be composed with 75 gallons of water, 4 lbs. of indigo, 5 lbs. of sulphate of iron, 5 lbs. of lime, and 1 lb. of soda. Begin to reduce the indigo into very fine powder, and to slack the lime; afterwards wash the powder in lye, and dissolve the sulphate of iron. This being done, put the water, indigo, lime, soda, and sulphate of iron in a deep kettle; shake the whole well; raise the temperature of the bath to 104 or 122°; maintain at this temperature the first two hours, then pass the stuffs. When, after using it, the bath begins to weaken, add to it 4 lbs. sulphate of iron, and 2 lbs. of quicklime so to dissolve the portion of indigo which by the contact with the air has oxygenized, and precipitated. It is only sometimes after this addition, that it is necessary to throw a new quantity of indigo.

The blue stone vat is a mixture of—

100 pails of water,
12 lbs. Potash or Soda,
4 " Bran,
4 " Madder.

The alkali, madder, and bran being diluted in water, boil some time; carry afterwards the liquor and residue into a kettle having a conical form, on a furnace; add the indigo well ground, and stir well. Cover the vat, and make fire round, and keep the bath at the temperature of 104 to 122°; shake the bath, and repeat that operation every twelve hours till ready to dye, which is ordinarily the case after 48 hours. The bath must be then of a fine yellow color covered with cupreous pastes, and a blue skim. When you dye, the bath becomes weaker because a great quantity of coloring matter is oxygenated, and precipitated. You can redissolve it in boiling a portion of the liquor of the vat in adding to it the quarter of the quantity of alkali, the quarter of the quantity of bran, and the quarter of the quantity of madder used primitively, and in pouring the mixture in the vat itself. When you ascertain that the indigo is exhausted, add a new quantity. It is evident that in this vat the deoxidizing agents are the bran and madder.

The woad vat is very similar to the above; it differs only by this, it enters a certain quantity of pastil and lime in its composition and no potash or soda. The quantity of matters to use are the following:—

1000 to 1125 gls. of Water,
415 lbs. of Pastil,
8 " " Woad,
2 " " Lime,
20½ " " Indigo.

1. Boil the water in a kettle for three hours with the woad, madder, and bran, take out the woad and transvase the liquid in a wooden vat in which you throw the pastil well divided.

This vat is about 8½ feet deep, and 6 feet in diameter; it is placed in a closed room and put in the ground.

During all the time you transvase and at least for $\frac{1}{4}$ of an hour after, you must shake all the matters contained in the bath to mix them well.

2. Cover exactly the vat and leave it 6 hours; shake again for $\frac{1}{2}$ an hour, repeat this operation every two or three hours till you perceive blue veins at the top; add the lime and immediately after, the powdered indigo; shake again the bath twice in the space of 6 hours and leave to settle; it takes a good yellow color; then you can pass the stuffs in.

3. From the moment the bath can be used it is necessary to throw in a pound of slacked lime and to heat it every two or three days so to keep it at the temperature of 95 to 122°. If you have no steam to heat, transvase the greatest part of the liquid in a kettle under which you make fire, and carrying this liquid in the vat, and covering it till you use it.

There is another method to prepare this vat. While the water passes in the vat, add to it about 155 lbs. of pastil shells partially softened in water; add to them 12 lbs. of indigo in fine powder with as less water as possible, and when very thick, stir well so to mix all the matters.

When the vat is full, powder its surface with 6¼ lbs. of good madder, 4 lbs. of slacked lime, and 4 quarts of bran; shut the vat; cover it with blankets, and leave it for six hours.

Stir the vat every three hours for half an hour every time till you perceive blue veins at the surface.

Stir again twice in the space of 6 hours, and at the end of the last stirring, throw at the surface from 7 to 8 ounces of lime. The stirring being done, cover the vat; three hours after, stir anew without adding lime, except if the fermentation is too quick; when this happens add at the end of the stirring 23 to 25 ounces of lime.

At this time the bath must be of a golden yellow color; the smell must not be too sharp or too sweet; ascertain again that the vat is in a good state when there appears at its surface *blue veins*, and a light skim of a fine blue color.

Stir then the vat every three hours till a specimen put for half an hour in it, is drawn off of a fine blue color, and takes quickly the blue color.

Stir again for the last time, and three hours after it is ready to work. The opening of the wool is done with 36 yards of woollen cloth or the same equivalent in weight of wool; leave it half an hour; wring out. If the wool was not dark enough, pass again once or twice according to the shade to be obtained.

Stir the vat, and put in it a little lime. To warm again the vat, transvase the $\frac{2}{3}$ of the vat in a kettle, and heat it till 197°; pass again the bath in the vat in stirring, and add in the same time from 2 to 4 lbs. of indigo, and from time to time a little pastil, bran, and madder; keep the vat well covered.

This vat is governed like the new vat, and well conducted, can last several years. When you do not use it, stir it at least twice a week.

Stuffs dyed blue must be washed carefully to carry away the parts not fixed on the wool, and for the dark blue it is best to press them in a little soap water which does not act on the blue.

Dyeing of Silk in Blue.

To dye the silk blue they use the woad vat described above; they put in, more indigo than the doses indicated, but the bran and madder are the same. The other vats cannot be used to dye silk because they do not dye quick enough.

When the vat is ready to use, add to it about two pounds of carbonate of soda, and $\frac{1}{8}$ of madder; shake the whole a quarter of an hour after it is ready to use.

Pour the silk in this bath, after cooking it with 30 per cent. of soap and washing it well in running water. As the silk does not take a smooth color easily, it is better to dye it by small portions, and when dyed, to air it; throw it in pure water and wring it out several times. The silk that you have dyed must dry very quick.

When the bath becomes weaker add one pound of sub-carbonate of soda, a little madder and a handful of well washed bran.

Indigo alone cannot dye the silk dark blue, it must be prepared in giving it another color or *bottom*. For the *Turkish Blue* they give first a very strong bath of archil; one less strong for the *King Blue*, afterwards they pass on a new vat. The other blues are done without bottom.

You can make a blue as dark as the king blue in using cochineal instead of archil, to give it more solidity; it is then called fine blue.

Dyeing stuffs blue with the soluble blue and distillated indigo.

Boil the wool one hour in a bath of cream tartar, 4 ounces by every pound of wool, raise, in the same bath put some soluble blue or distillated indigo previously dissolved in water. Boil till the required shade.

On wool and silk tissues give a boiling with cream tartar and alum, and dye as wool but without boiling.

For the silk, mordant tepid, and in this bath put a certain quantity of soluble blue according to the shade to be obtained.

According to Mr. Chevreul, you can dye blue, 20 lbs. of wool by the following process:—

Mordant at 158° with—

2 lbs. 10 ounces of Alum,
22½ ounces of Cream Tartar.

Handle the wool in for half an hour; raise; give air, and add in the bath more or less carmine of indigo dissolved according to the shade, and work the wool in till the required color. If you wish to obtain a violaceous lilac with the carmine, add a little ammoniacal cochineal.

Logwood Blue.

These blues are not as solid as those obtained by indigos or prussiate of iron. This dye is done like the Brazil red if it is that they add to the bath some verdigris or alkali. For one pound of mordanted wool use—

1¾ ounces of Wood,
15 to 20 pints of Water,
¾ ounces Verdigris.

They use also Logwood and verdigris to remount a light bottom of solid or vat blue, but those kinds of blues are not solid.

CHAPTER XVII.

ASSAY OF THE COLORS.

Assay of the Blues.

THERE are four kinds of blues, viz:—

Indigo,
Prussian Blue,
Logwood,
Ultramarine.

Indigo has for generic characteristic to be destroyed by heat without residue and to be decolorized by chlorine, nitric acid.

The *soluble blue* is not alterable by potash.

The *Saxony blue* disappears by potash, but can be re-established by the action of an acid.

The *Prussian blue* has for generic characteristic to be destroyed by heat in leaving a residue of peroxide of iron; it is not attacked by chlorine but decolorized by potash.

The *Logwood blue* when touched by an acid turns red, is decolorized by heat, and leaves on the cloth a brownish residue of alumina and oxide of iron; dissolved in nitric acid, the ash gives a liquor which turns blue by ammonia.

Ultramarine is rarely employed; it is ascertained by its shade and its unalterability by the fire. Hydrochloric acid decolorizes it, nitric acid decolorizes it completely.

The *mixed blue* Prussian and Saxony are detected by chlorine or nitric acid, which destroys the second and leaves the first intact.

Assay of the Reds.

All reds, except the saffron rose, which is destroyed by chlorine and by heat without residue, belong to the colors which are the result of the combination of a mordant of alumina or alumina and oxide of tin with a coloring matter.

Their general character is to be destroyed by chlorine, to leave a residue by incineration. They can be divided into three classes:—

Red formed by Madder and derivatives,
" " " *Cochineal,*
" " " *Wood.*

Treated by hydrochloric acid the red formed by madder turns yellow or yellow orange; thus modified, if they are dipped in a milk lime bath, all places touched by acids take a fine violet shade, which becomes rose in passing them in soap water.

The reds by cochineal and wood, by acid take a current shade, passed in lime water they form a violet which disappears in soap.

Concentrated sulphuric acid turns the cochineal into a bright cherry red, and the wood into yellow orange.

Assay of Yellows.

They are distinguished into *woad*, *quercitron*, *fustic*, *turmeric*, *astringent substances*, *annotto*, *chrome*, *orpiment*, *nankin*, *and rust*.

Yellows with *quercitron* are destroyed by chlorine, but do not turn sensibly orange by alkalies; chloride of tin, nitric acid give them a reddish color.

Fustic Yellows are destroyed by chlorine. Potash turns them Turkish yellow; treated by chloride of tin they pass orange. Treated by nitric acid they take a dust color. Orange or Nankin by fustic turn red by sulphuric acid, and catechu shade by potash; they are destroyed by nitric acid.

Turmeric yellows are decolorized by chlorine, and turn red orange by alkalies.

Sumach yellows take a lighter shade by chloride of tin; redden by nitric acid.

The orange yellow by *annotto* are attacked with difficulty by chlorine; they pass greenish blue by sulphuric acid; they take a dark shade, and disappear by nitric acid.

Chrome yellows are not destroyed by heat; they are not attacked by weak hydrochloric acid, but destroyed by concentrated hydrochloric acid; they are dissolved and decolorized by caustic potash; they are transformed in orange when dipped in boiling lime water.

Orpiment yellows are not attacked by hydrochloric acid, soluble in potash, destroyed by nitric acid.

Nankins and *Rusts* give a residue by incineration; chlorine is without action on them; hydrochloric acid attacks them. A mixture of hydrochloric acid and chloride of tin reduces immediately the rust, and makes it appear white all places it touches. Hydrochloric acid and prussiate of potash applied on the rust producc a blue color.

Assays of Green.

Greens are divided into four kinds, viz:—

Green with Indigo,
" " *Prussian Blue,*
" " *Vegetal Coloring Matters,*
" " *Mineral Salts.*

Greens with *indigo* are destroyed by heat without leaving any other residue than the one furnished by the yellow alone, and they are destroyed by chlorine in leaving the yellow.

With *Prussian blue* the color is not destroyed by chlorine, but is attacked by potash, which destroys the blue and yellow.

With *vegetable coloring matters*, the blue is destroyed by chlorine; with logwood it reddens by acids.

The green with *metallic salts*, such as arsenite of copper, turns yellow by hydrochloric acid, and passes blue by ammonia.

Assay of the Violets.

Violets with madder leave by incineration a residue of peroxide of iron, and are decolorized by chlorine; treated by hydrochloric acid, they take a dirty orange color, passed in a milk of lime. All parts touched by acids take a violet blue shade, that they keep in a bath of boiling soap.

Violets with logwood, when incinerated, leave a white ash, are destroyed by chlorine; treated by hydrochloric acid they turn red.

Violets with cochineal leave always an iron residue when incinerated.

Violets with orkanet are slightly attacked by chlorine; they do not turn red by nitric or hydrochloric acids; they turn blue by potash.

CHAPTER XVIII.

COMMERCIAL ASSAYS OF INDIGO.

Process by Immediate Analysis.

HAVING occasion to examine some commercial indigos, I was struck by the great variation which existed in the different methods of assay, and not being sure of the best process, I resolved to try which was the quickest and most exact method. Having at my disposition a great many kinds of indigo, my researches were more easy.

I examined first what were the constituent principles contained in the indigos, and what were the quantities, and at last I weighed the coloring matter by different methods, in order to ascertain which was the best; this work has engaged me nearly one year.

Research of the Immediate Principles.

In the search of the immediate principles contained in indigos, I have followed the process indicated by Mr. Chevreul,[1] and have arrived at the same results, only with variation in the proportions. I think proper to recall succinctly the method of proceeding with success.

Indigos were treated, 1st, by water; 2d, by alcohol; 3d, by hydrochloric acid.

Action of Water.

Dry indigo in fine powder is treated by water for 12 hours at a gentle heat (86° to 104°); decant the liquor, and filter; continue

[1] Ann. de Chim. et de Phys. Vol. 66, page 5.

thus till the indigo is completely exhausted. I distil the liquids which give me an ammoniacal water, and I finish the evaporation in a porcelain dish; by evaporation the liquid becomes covered with a blue skim; at the end of the evaporation greenish flakes are precipitated, and they are separated by decantation.

Once the washing waters in syrupy consistence, I add alcohol at 95°, and filter; the liquor has a red scarlet color; I diluted with water, and heated. It disengages ammonia: weak acids turn it green; concentrated acids precipitate a green matter.

Water dissolves—

Ammonia,
White Indigo,
Green Matter,
Gum,
Extractive Matter (small quantity).

Action of Alcohol.

Indigo exhausted by water was dried, and treated by alcohol; the first washings are red, the following are purple violet, and at last blues. I concentrated them; they have a blackish-red residue, which is treated by warm water. It takes a yellowish-green color, and turns red by alkalies; this phenomenon is due to the green matter. The residue of the evaporation well washed, treated by cold alcohol becomes of a purple red; it dissolves a green resin.

The matter insoluble in cold alcohol is blue, and has all the properties of indigo.

Alcohol then dissolves—

Green Matter,
Red Resin,
Indigo.

Action of Weak Hydrochloric Acid.

Weak hydrochloric acid takes to indigo—

Oxide of Iron,
Alumina,
Carbonate of Lime,
Red Resin.

The residue consist in—

Silica,
Indigo.

It is incinerated, and the ashes represent the *Silica.*

I operated on 38 varieties of indigo, viz: 9 *Java*, 7 *Bengalis*, 5 *Caracas*, 3 *Guatemala*, 3 *Madras*, 3 *Manilla*, 4 *Bombay*, 1 *Philippines*, 3 *Polygonum Tinctorium.*

The results obtained are given in the following table.

Table of the Elementary Composition of different Indigos.

Nature of the Indigo.	Matters Soluble in Water.		Matters Soluble in Alcohol.		Matters Soluble in Hydrochloric Acid.			Insoluble Matters.		Indigo, per cent.
	Ammonia, green matter, extractive, gum.	Indigo.	Green matter, green resin.	Indigo.	Red resin.	Carbonate of lime.	Oxide of iron, Alumina.	Silica.	Indigo.	
Java.										
Surfine	0.7	4.0	0.5	12.0	1.3	0.2	0.3	1.0	80.0	96.0
Purple	1.2	4.0	4.3	10.0	1.5	1.0	1.0	2.0	75.0	89.0
Fine	4.0	5.0	4.5	15.0	1.5	0.5	0.5	1.0	68.0	88.0
Surfine purple	4.3	4.0	6.2	10.0	2.5	1.0	0.5	1.5	70.0	84.0
Surfine violet	6.0	3.0	8.0	15.0	4.0	2.0	2.0	4.0	56.0	74.0
Fine blue	7.7	4.0	10.3	15.0	4.0	1.5	0.5	3.0	54.0	73.0
Fine violet	9.0	2.0	7.8	12.0	3.2	3.0	2.5	3.5	57.0	71.0
Fine	7.5	6.0	10.5	12.0	2.5	3.5	2.0	3.0	53.0	71.0
Blue-black	9.7	2.0	21.3	10.0	8.0	2.5	4.5	8.0	34.0	56.0
Bengalis.										
Surfine purple	0.5	4.0	0.5	14.0	2.0	0.3	0.7	1.0	77.0	95.0
Fine violet	5.5	5.0	6.5	11.0	1.0	0.5	0.5	1.0	69.0	85.0
Surfine violet	7.5	3.5	3.0	10.0	2.5	3.0	1.0	2.0	68.5	82.0
Fine violet	7.0	4.0	6.5	14.0	5.5	3.0	2.0	2.0	56.0	74.0

Table of the Elementary Composition of different Indigos—Continued.

Nature of the Indigo.	Matters Soluble in Water.		Matters Soluble in Alcohol.		Matters Soluble in Hydrochloric Acid.			Insoluble Matters.		Indigo, per cent.
	Ammonia, green matter, extractive, gum.	Indigo.	Green matter, green resin.	Indigo.	Red resin.	Carbonate of lime.	Oxide of iron, Alumina.	Silica.	Indigo.	
Bengalis—*Continued.*										
Fine violet red	6.0	5.0	8.5	12.0	5.5	2.0	1.0	2.0	58.0	75.0
Violet	9.0	4.5	8.0	9.0	7.0	3.0	2.0	5.0	52.5	66.0
Low cupreous	10.0	1.0	25.0	9.0	8.0	4.0	3.0	5.0	35.0	45.0
Caracas	6.5	5.0	8.5	12.0	2.0	0.5	0.5	1.0	64.0	81.0
Idem	7.5	4.0	9.5	9.0	3.0	1.5	1.5	2.0	62.0	75.0
Idem	8.0	3.0	8.0	10.0	5.0	2.0	3.0	8.0	53.0	66.0
Idem	10.0	2.0	12.0	7.0	8.0	4.0	2.0	5.0	50.0	59.0
Idem	14.5	1.5	13.0	8.0	5.5	4.0	3.0	6.0	46.5	56.0
Guatemala.										
Kurpath	5.0	4.0	10.0	10.0	3.0	1.0	1.5	1.5	64.0	78.0
Blue	9.0	4.0	11.0	9.0	4.5	3.5	1.0	3.0	55.0	68.0
Violaceous	13.0	2.0	14.0	8.0	6.0	4.0	2.0	7.0	44.0	54.0
Madras	12.0	3.5	12.5	9.0	6.5	4.0	3.0	4.0	45.5	58.0
Idem	15.0	2.0	16.5	8.0	9.5	5.0	5.0	7.0	32.0	42.0
Idem	12.0	1.5	22.0	3.0	6.0	7.0	7.0	14.0	27.5	32.0

Table of the Elementary Composition of different Indigos—Concluded.

Nature of the Indigo.	Matters Soluble in Water.		Matters Soluble in Alcohol.		Matters Soluble in Hydrochloric Acid.			Insoluble Matters.		Indigo, per cent.
	Ammonia, green matter, extractive, gum.	Indigo.	Green matter, green resin.	Indigo.	Red resin.	Carbonate of lime.	Oxide of iron, Alumina.	Silica.	Indigo.	
MANILLA.										
Blue	12.5	4.0	10.5	8.0	9.0	3.0	5.0	10.0	38.0	50.0
Dark	15.0	4.0	15.0	7.0	10.5	4.0	6.5	7.0	31.0	42.0
Very dark	16.0	3.5	20.0	8.0	9.0	5.0	4.0	6.0	32.5	40.0
BOMBAY.										
Light blue	15.0	2.5	25.0	4.0	10.0	5.0	4.0	6.0	28.5	35.0
Tarnish	10.0	1.0	25.0	2.0	12.0	8.0	5.0	9.0	28.0	31.0
Spotted	10.0	1.0	20.0	4.0	10.0	15.0	5.0	11.0	24.0	29.0
Brown-black	12.0	2.5	25.0	3.0	13.0	10.0	2.0	9.0	21.5	27.0
PHILIPPINES	10.0	5.0	20.0	4.0	8.0	9.0	3.0	7.0	34.0	43.0
INDIGO OF THE POLYG. TINCTORIUM	11.0	5.5	21.0	5.0	6.0	7.0	2.0	10.0	32.5	43.0
Idem	15.0	1.5	22.0	2.0	11.0	8.0	3.0	13.0	24.5	28.0
Idem	15.0	1.0	22.5	2.5	14.5	8.0	6.0	20.0	10.5	14.0

CHAPTER XIX.

COMMERCIAL ASSAYS OF INDIGO.

Estimation of Water and Ashes.

THE estimation of water and ashes is a very important operation; for it is often the case that indigos of first quality are damaged by sea water, and give them a more elevated weight than it is in reality; by the estimation of ashes it gives approximately the quantity of coloring matter contained in the indigo.

Estimation of Water.

Take from 15 to 24 grains of indigo in fine powder; put it in a porcelain dish, and heat it at 220° in an oven; leave it till completely dry, *i. e.*, till the weight does not change.

The following table gives the quantity of water contained in each indigo.

Table indicating the quantity of Water contained in Indigos.

Nature of the Indigo.	Water per cent.
JAVA.	
Surfine	2.5
Purple	2.8
Fine	2.9
Surfine Purple	2.1
" Violet	2.5
Fine Blue	2.6
" Violet	3.8
Fine	4.9
Blue Black	3.7
BENGALIS.	
Surfine Purple	5.4
Fine Violet	1.3
Surfine "	6.1
Fine "	2.2
Fine Violet Red	2.3
Violet	2.3
Low Cupreous	2.7

Nature of the Indigo.	Water per cent.
CARACAS	5.0
Idem	5.0
Idem	5.3
Idem	4.8
Idem	4.7
GUATEMALA.	
Kurpath	2.8
Blue	2.7
Violaceous	1.9
MADRAS	3.4
Idem	3.7
Idem	3.5
MANILLA.	
Blue	5.8
Dark	5.9
Very Dark	5.5
BOMBAY.	
Light Blue	4.8
Tarnish	4.7
Spotted	4.7
Brown Black	3.2

Nature of the Indigo.	Water per cent.
PHILIPPINES	6.5
Indigo of the Polygonum Tinctorium	5.5
Idem	5.9
Idem	4.5

We see by the above numbers that the quantity of water depases rarely 6 per cent.

The Java contains in mean			. . .	3.08
Bengalis	"	"	" . . .	3.18
Caracas	"	"	" . . .	4.96
Guatemala	"	"	" . . .	2.46
Madras	"	"	" . . .	3.53
Manilla	"	"	" . . .	5.73
Bombay	"	"	" . . .	4.35
Polygonum	"	"	" . . .	5.30

Determination of Ashes.

Having thus valuated the quantity of water, I determined by an incineration the quantity of ashes and organic matters contained in each indigo; for that I take 15½ grs. of dry indigo;

I heat it first slowly in a platina dish, and terminated the calcination in a reverberatory furnace.

The following table gives the quantity of ashes and organic matters contained in the indigo.

Organic Matters and Ashes contained in Indigos.

Nature of Indigo.	Organic Matters.	Ashes.
JAVA.		
Surfine	98.4	1.6
Purple	96.5	3.5
Fine	98.0	2.0
Surfine Purple . . .	97.0	3.0
" Violet . . .	92.5	7.5
Fine Blue . . .	95.0	5.0
" Violet . . .	93.5	6.5
Fine	91.5	8.5
Blue Black . . .	86.0	14.0
BENGALIS.		
Surfine Purple . .	98.0	2.0
Fine Violet . . .	98.5	1.5
Surfine Violet . . .	94.5	5.5
Fine " . . .	93.0	7.0
" " Red . .	95.0	5.0
Violet	91.0	9.0
Low Cupreous . . .	88.0	12.0

Nature of Indigo.	Organic Matters.	Ashes.
Caracas	98.0	2.0
Idem	95.0	5.0
Idem	87.5	12.5
Idem	89.0	11.0
Idem	87.0	13.0
GUATEMALA.		
Kurpath	96.0	4.0
Blue	92.5	7.5
Violaceous	87.0	13.0
MADRAS	90.5	9.5
Idem	84.5	15.5
Idem	73.0	27.0
MANILLA.		
Blue	83.0	17.0
Dark	83.5	16.5
Very Dark	85.0	15.0
BOMBAY.		
Light Blue	85.0	15.0
Tarnish	80.0	20.0
Spotted	70.0	30.0
Brown Black	79.0	21.0

Nature of Indigo.	Organic Matters.	Ashes.
PHILIPPINES	81.5	18.5
Indigo of the Polygonum Tinctorium . . .	82.0	18.0
Idem	78.0	22.0
Idem	70.0	30.0

We see that by an incineration alone it is possible to ascertain nearly the nature of an indigo; the more earthy matters it contains, the less rich it is in coloring matter. The greater part of the earth can be attributed to that attached to the plant, and introduced mechanically during the operation of the manufacture of indigo. We see that the indigo contains a mean of—

The Java . . .	5.73	per cent. of ashes.
Bengalis . .	7.00	"
Caracas . .	8.70	"
Guatemala . .	8.16	"
Madras . .	17.30	"
Manilla . .	16.10	"
Bombay . .	21.50	"
Polygonum .	23.30	"

These preliminary assays being done, I pass immediately to the estimation of the coloring matter by the different processes.

CHAPTER XX.

COMMERCIAL ASSAYS OF INDIGO.

Estimation by the Vat.

THE processes of weighing the coloring matters are numerous; hence so many errors. I have endeavored to ascertain by trying all the methods, which is the best, and it is those different experiments I am presenting now.

Estimation by Precipitation of Indigotine.

1ST PROCESS.—Take 155 grains of dried and powdered indigo; reduce it into a paste and introduce it into a porcelain dish with 310 grains of sulphate of iron and 2 quarts of water; boil a few minutes. Add to it from $1\frac{1}{4}$ to $1\frac{2}{3}$ ounces of caustic soda; shake well; cover the dish, and leave it to settle.

When all is deposited, decant carefully the liquid; on the deposit pour $1\frac{1}{2}$ quart of water; boil ten minutes; leave to settle, and decant anew.

To the deposit add 155 grains of sulphate of iron, 310 grains of caustic soda; boil; leave to settle, and decant.

Repeat the operation as long as the liquid colors in the air.

When the liquid does not color, add to the decanted water some hydrochloric acid; leave to settle, and decant.

Throw the deposit on a dried and weighed filter.

Wash the precipitate with water till it passes pure.

Dry it at 248°; weigh it. Its weight indicates the quantity of coloring matter.

2D PROCESS.—Operate the same as above, only to have less liquid, use—

Indigo	$15\frac{1}{2}$ grains.
Sulphate of Iron . .	31 "
Soda	62 to 77 grains.
Water	1 pint.

3D PROCESS.—The two above processes on account of the length of time occupied, can be modified in the following manner:—

Treat the indigo at the temperature of 194° by soda and sulphate of iron.

Introduce the whole in a ground stoppered bottle.

Heat several hours in a water bath; leave to settle; decant a certain quantity of liquor in a graduated glass; precipitate the indigotine by hydrochloric acid; filter; wash and dry at 248°, weigh.

I always operate on $15\frac{1}{2}$ grains of indigo, and 4 pints of water, and decant only 3 pints of liquid.

I suppose that in 3 pints I found 7.75 grains of indigotine. I obtain the total quantity by the following equation:—

$$3 : 7.75 :: 4 : x = \frac{7.75 \times 4}{3} = 10.33 = 66.64 \text{ per cent.}$$

The numbers obtained by the three processes are given in the following table.

Table indicating the Proportions of Indigotine in 100 Parts of Indigo.

Nature of Indigo.	First process.	Second process.	Third process.
JAVA.			
Surfine	94.5	95.0	95.5
Purple	87.0	87.7	88.9
Fine	87.0	87.5	88.0
Surfine purple	82.0	82.6	83.8
Surfine violet	72.0	72.4	73.9
Fine blue	71.5	72.6	72.8
Fine violet	69.9	70.0	70.9
Fine	69.9	70.0	70.9
Blue-black	54.0	54.9	55.8
BENGALIS.			
Surfine purple	93.5	94.0	94.9
Fine violet	83.0	84.3	84.9
Surfine violet	80.5	81.2	81.8
Fine violet	72.0	72.4	73.9
Fine violet red	73.0	74.4	74.8
Violet	64.5	64.9	65.7
Low cupreous	44.0	44.8	45.1
CARACAS	79.0	80.8	81.0
Idem	73.0	74.4	74.9
Idem	64.4	64.9	65.9
Idem	57.5	58.0	59.0
Idem	54.0	54.9	55.9
GUATEMALA.			
Kurpath	75.8	76.2	77.9
Blue	65.0	66.6	67.8
Violaceous	52.2	53.4	54.0
MADRAS	55.4	56.6	57.9
Idem	40.9	41.1	42.1
Idem	30.0	30.9	31.9
MANILLA.			
Blue	48.7	49.2	49.8
Dark	40.9	41.2	42.0
Very dark	39.0	39.1	39.9

Table indicating the Proportions of Indigotine in 100 Parts of Indigo—Concluded.

NATURE OF INDIGO.	First process.	Second process.	Third process.
BOMBAY.			
Light blue	33.7	34.3	34.9
Tarnish	30.0	30.8	31.1
Spotted	27.4	27.9	28.8
Brown-black	25.2	26.4	27.1
PHILIPPINES	42.0	42.3	43.2
POLYGONUM TINCTORIUM	42.0	42.4	43.0
Idem	25.4	25.9	27.9
Idem	11.5	12.4	14.1

We see that the first process gives too weak numbers; the second gives some more elevated, and at last the third is the nearer to the numbers found by immediate analysis.

By the first process, whatever was the care taken, it was impossible to exhaust completely the vat, and there remains always a certain quantity of coloring matter in the precipitate which stays undissolved, and occasions a more or less lost, and besides operating on a large quantity of liquid, there is always some lost during the filtrations.

The second process presents the same inconveniences, but at a less degree, the quantity of matters used being less.

In the third process we operate on a determined quantity of liquid; the difficulty is removed, for all the indigo is in solution in the liquid; we have then an homogeneous liquor, and if it is a lost, it is only during the filtrations.

For persons wishing to make analysis of indigo by the vat process, we recommend them the third.

Estimation by Precipitation of the Coloring Matter on Woollen Threads.

This process is very difficult, and requires the sure eye of the dyer to judge of the richness of the coloring matter, and besides, it requires a great habit of manipulations, but its advantages are that it permits to judge of the beauty of the color.

For a chemist this process is impracticable, for he will always find numbers lower than the reality, but it is a good method for a dyer, who without telling exactly the richness of an indigo, would ascertain easily its quality.

To execute this process prepare two vats, one with pure indigotine, the other with the indigo to assay. Employ the following proportions:—

	With Indigotine.	With Indigo.
Coloring matter .	15½ grs.	15½ grs.
Sulphate of Iron .	54¼ "	54¼ "
Potash . . .	54¼ "	54¼ "
Water . . .	1 quart.	1 quart.

The bottles are entirely full; leave to rest ½ an hour, and dip in it 31 grains of woollen thread; leave it 10 minutes, and make the same with pure indigotine.

This assay is little satisfactory on account of the difficulty in exhausting completely the vat in handling in skeins of wool of an equal weight, and it is difficult to compare the quantity of dye wool to the one which shall be dyed in using indigotine the same manner. The relative value of the color only can be judged, and you see if the indigo will give a fine color.

Nevertheless, in the following table I have given the results obtained.

Three skeins of wool weighing 31 grains each, have been sufficient to exhaust a vat in having each skein in it five minutes—the gam was from 30 shades. With pure indigotine I have had—

31 *grains of wool to the shade* .	28
31 " " " " . .	15
31 " " " " . .	5

Table indicating the Proportion of Indigotine in 100 Parts of Indigo by Dyeing with the Vat.

NATURE OF INDIGO.	1ST EXPT. 31 grs. wool to the shade.	2D EXPT. 31 grs. wool to the shade.	3D EXPT. 31 grs. wool to the shade.	Indigo, per cent.
INDIGOTINE	28.0	15.0	5.0	100
JAVA.				
Surfine	26.0	14.0	4.5	93
Purple	24.0	13.0	4.3	86
Fine	23.5	12.5	4.2	84
Surfine purple . .	22.4	12.0	4.0	80
Surfine violet . .	19.5	10.5	3.5	70
Fine blue . . .	19.3	10.3	3.4	69
Fine violet . . .	18.7	9.8	3.3	67
Blue	18.7	9.8	3.3	67
Blue-black . . .	14.5	5.9	2.6	52
BENGALIS.				
Surfine purple . .	25.5	13.6	4.5	91
Fine violet . . .	22.6	12.1	4.0	81
Surfine violet . .	22.0	11.8	3.9	79
Fine violet . . .	20.1	10.8	3.6	72
Fine violet red . .	20.0	10.0	4.0	73
Violet	15.5	9.0	3.0	62
Low cupreous . .	14.5	6.0	2.0	41
CARACAS	22.0	11.5	4.0	78
Idem	20.0	11.0	3.0	71
Idem	17.5	9.0	3.0	62
Idem	16.0	8.0	2.3	55
Idem	15.0	7.8	2.0	52
GUATEMALA.				
Kurpath . . .	21.0	12.0	3.0	75
Blue	18.0	9.0	3.0	63
Violaceous . . .	14.0	8.0	2.0	51
MADRAS	16.0	8.3	2.0	55
Idem	11.0	6.0	2.0	40
Idem	8.4	5.0	1.0	30

Table indicating the Proportion of Indigotine in 100 Parts of Indigo by Dyeing with the Vat—Concluded.

NATURE OF INDIGO.	1st Expt. 31 grs. wool to the shade.	2d Expt. 31 grs. wool to the shade.	3d Expt. 31 grs. wool to the shade.	Indigo, per cent.
MANILLA.				
Blue	14.0	7.0	2.0	48
Dark	11.0	6.0	2.0	40
Very dark . . .	10.5	5.7	1.9	38
BOMBAY.				
Light blue . . .	8.9	4.8	1.6	32
Tarnish	7.8	4.2	1.4	28
Spotted . . .	7.0	3.7	1.2	25
Black-brown . .	6.7	3.6	1.2	24
INDIGO OF PHILIPPINES .	10.0	6.0	2.0	40
INDIGO OF POLYG. TINCT. .	10.0	6.0	2.0	40
Idem	7.0	3.7	1.2	25
Idem	3.3	1.8	Near 1	12

In examining the above table we see that the results have no reliable exactness, and that the numbers are far from those obtained by the other methods. Thus the *Java surfine* by this process gives 93 per cent. of coloring principle, while by the immediate analysis it gives 96;

by the precipitation of indigotine from a vat it gives 94.5, 95, 95.5; but while I have a difference of near 3 per cent. the eye of a dyer recognizes an indigo of first quality.

CHAPTER XXI.

COMMERCIAL ASSAYS OF INDIGO.

Assay by the Sulphuric Dissolution of Indigo.

THE following processes are more employed because they are more simple and more rapid; they are more exact, and with a little practice you could succeed in a short time in performing them well.

(*a.*) *Assay by Dyeing.*

Take a little glass vial in which you introduce 15½ grains of the indigo to try, in fine powder and dried; pour on it ½ an ounce of sulphuric acid at 66°.

Repeat the same operation with 15½ grains of indigotine. Heat for 6 hours at 149°; dilute with water, and afterwards add enough of this liquid to make 2 quarts or 2000 cubic centimeters, which contain 15½ grains of indigotine, and 20 cubic centimeters contain 0.155 grains.

Take 20 cubic centimeters of each dissolution, and pour in, a skein of wool weighing 15½ grains; leave it 24 hours, and repeat the operation till the bath is exhausted.

The shades are compared to a gam of thirty shades.

With pure indigotine—

15½ grains of wool is dyed to the shade 20 after 24 hours.

15½ grains of wool is dyed to the shade 7 after 24 hours.

15½ grains of wool is dyed to the shade 3 after 24 hours.

Then it wants 46 grains of wool to take 0.155 grains of indigotine.

Repeat the same operation with the indigo to try, and by the way of proportions, you will find the quantity of coloring matter contained in each kind.

The numbers obtained are given in the following table.

Table indicating the Proportion of pure Indigo contained in 100 Parts of Indigo by Dyeing with the Sulphate of Indigo.

NATURE OF INDIGO.	15.5 wool is dyed to the shade.	15.5 wool is dyed to the shade.	15.5 wool is dyed to the shade.	Indigo, per cent.
JAVA.				
Surfine	19.0	6.5	2.8	95
Purple	17.5	6.0	2.5	88
Fine	17.0	6.0	2.0	86
Surfine purple	16.0	5.5	2.0	80
Surfine violet	14.5	5.5	2.0	73
Fine blue	14.3	5.0	2.0	72
Fine violet	14.0	4.9	2.0	70
Fine	14.0	4.9	2.0	70
Blue-black	11.4	4.0	1.5	57
BENGALIS.				
Surfine purple	18.8	6.5	2.8	94
Fine violet	16.6	5.8	2.5	83
Surfine violet	16.0	5.5	2.4	80
Fine violet	14.5	5.0	2.0	72
Fine violet red	14.8	5.0	2.0	74
Violet	12.5	4.3	1.5	63
Low cupreous	8.6	3.0	1.0	43
CARACAS	16.0	5.5	2.4	80
Idem	14.5	5.0	2.0	73
Idem	12.8	4.5	1.9	64
Idem	11.5	4.0	1.5	57
Idem	10.5	3.8	1.5	54
GUATEMALA.				
Kurpath	15.0	5.0	2.0	75
Blue	13.0	4.5	1.9	65
Violaceous	10.0	3.5	1.5	50
MADRAS	11.0	3.9	1.5	55
Idem	8.0	2.9	1.0	40
Idem	6.0	2.0	Near 1	30

Table indicating the Proportion of pure Indigo contained in 100 *Parts of Indigo by Dyeing with the Sulphate of Indigo*—Concluded.

NATURE OF INDIGO.	15.5 wool is dyed to the shade.	15.5 wool is dyed to the shade.	15.5 wool is dyed to the shade.	Indigo, per cent.
MANILLA.				
Blue	9.5	3.2	1.5	40
Dark	8.0	2.9	1.0	40
Very dark . . .	7.5	2.5	1.0	38
BOMBAY.				
Light blue . . .	6.5	2.5	1.0	34
Tarnish . . .	6.0	2.0	1.0	30
Spotted . . .	5.5	2.0	Near 1	28
Brown-black . .	5.0	1.0	1.0	25
INDIGO OF PHILIPPINES .	8.0	2.5	1.5	40
INDIGO OF THE POLYG. TINCT.	8.0	2.5	1.5	40
Idem	5.0	1.0	1.0	25
Idem	2.5	1.0	Far from 1	13

This process is long and does not give very exact results. The following is better.

(*b*.) *Assay by the Colorimeter.*

The use of the colorimeter is due to Mr. Houton Labillardiere. It is a very useful instrument, which permits to see in a few minutes the richness of a coloring matter. This apparatus is too well known to be described here. I operated as follows:—

At 104° I dissolve 15½ grains of pure indigotine in ⅔ ounce of sulphuric acid; after the dissolution is operated I add water so to form 1 quart of liquid; I repeated the same operation on every indigo to try.

In one of the tubes of the colorimeter I introduce 10 cubic centimeters of solution of indigotine, and in the other 10 cubic centimeters of the dissolution of the indigo to try. I add water to the indigotine till the shade of the dissolution is similar to that of the indigo. The proportion of water added indicates the proportion of the coloring matter.

The numbers obtained are found in the following table:—

Table representing the Value of Indigos tried by the Colorimeter.

Nature of the Indigo.	To 10 c.c. of dissolution of Indigotine you have to add.	Indigo per cent.
JAVA.		
Surfine	0cc4 water	96
Purple	1.1	89
Fine	1.2	88
Surfine Purple	1.6	84
" Violet	2.6	74
Fine Blue	2.7	73
" Violet	2.9	71
Blue	2.9	71
Blue Black	4.4	56
BENGALIS.		
Surfine Purple	0.5	95
Fine Violet	1.5	85
Surfine Violet	1.8	82
Fine "	2.6	74

Nature of the Indigo.	To 10 c. c. of dissolution of Indigotine you have to add.	Indigo per cent.
BENGALIS.		
Surfine Violet Red	2.5 water	75
Violet	3.4	66
Low Cupreous	5.5	45
CARACAS	1.9	81
Idem	2.5	75
Idem	3.4	66
Idem	4.1	59
Idem	4.4	56
GUATEMALA.		
Kurpath	2.2	78
Blue	3.2	68
Violaceous	4.6	54
MADRAS	4.2	58
Idem	5.8	42
Idem	6.8	32

Nature of the Indigo.	To 10 c.c. of dissolution of Indigotine you have to add.	Indigo per cent.
MANILLA.		
Blue	5.0 water	50
Dark	5.8	42
Very Dark	6.0	40
BOMBAY.		
Light Blue	6.5	35
Tarnish	6.9	31
Spotted	7.1	29
Brown Black	7.3	27
INDIGO OF PHILIPPINES	5.7	43
POLYG. TINCT.	5.7	43
Idem	7.2	28
Idem	8.6	14

This process has an advantage over all the others, it is simple, rapid, and very exact; with a little use it is preferable to all others even the following. I always use and recommend it.

CHAPTER XXII.

COMMERCIAL ASSAYS OF INDIGO.

Assay by Hypochloride of Lime.

INDIGO being dissolved, I determine the proportion of coloring principle that contains the dissolution by the quantity of chlorine that requires a given volume to be decolorized. When applied the process does not present always the required exactness. If the acid used for the dissolution of indigo contains sulphurous acid, or if some is formed accidentally, some coloring matter is destroyed, and it is a cause of error which gives less coloring matter than really exists. The unequal manner by which indigo is decolorized, the manner, more or less imperfect, by which the dissolution is operated, are all causes of error, the variations which have occurred in the proof liquor can be the cause of some errors.

You can remedy all these imperfections in operating in the following manner, and treating comparatively pure indigotine and dry indigo:—

Weigh 15½ grs. of each; dissolve them in half an ounce of sulphuric acid of Nordhausen perfectly free from sulphurous acid; leave the mixture 24 hours at 122°, being careful to avoid the formation of sulphurous acid; dissolve the solution in one quart of water.

There are, then, two ways to operate:—

1st. Take 100 c. c. of the solution of indigo and search the number of cubic centimeters of a solution of hypochloride of lime necessary to decolorize. The richness of the coloring principle is proportional to the number of cubic centimeters of chlorine absorbed.

2d. Take 5 c. c. of hypochloride of lime and search the volume of sulphate of indigo decolorized, the richness is in inverse ratio of the volume of decolorized indigo.

The way to operate in this case is the following:—

Take $2\frac{1}{2}$ c. c. of hypochloride of lime at $1\frac{1}{2}°$ B.; pour it in a vase; introduce in it 50 c. c. of sulphate of indigo. If the liquid turns yellow immediately, it is an excess of chlorine; if indigo predominates add hypochloride of lime till in excess, then with a graduated glass, add of indigo till the dissolution becomes green.

$2\frac{1}{2}$ c. c. of hypochloride of lime decolorize 50 c. c. of normal solution of pure indigotine, it is then easy to calculate the quantity of coloring principle, while it is in inverse ratio of the volume of the decolorized dissolution.

A = *the number of c. c. of the normal dissolution.*

B = *the number of c. c. of the Indigos.*

We have then—

$$B : A :: 100 : x = \frac{A + 100}{B} = x.$$

This process is as exact as the first, but is languider; it requires much practice to arrive to a perfect discoloration, while by the first you see immediately the term of the operation.

I have always used the first, and it has given me the same results as the second. The numbers obtained are given in the following table:—

Nature of Indigos.	Number of cubic centimeters of hypochlorite of lime to discolorize 100cc of dissolution.	Indigo per cent.
PURE INDIGOTINE . .	120c	100
JAVA.		
Surfine . . .	115.2	96
Purple . . .	106.8	89
Fine . . .	105.6	88
Surfine, Purple .	100.8	84
" Violet .	88.8	74
Fine Blue . .	87.6	73
" Violet . .	85.2	71
Fine . . .	85.2	71
Blue Black . .	67.2	56
BENGALIS.		
Surfine Purple .	112.8	94
Fine Violet . .	99.6	83
Surfine Violet . .	98.4	82
Fine " . .	87.6	73
Fine Violet Red .	90.0	75
Violet . . .	78.0	65
Low Cupreous . .	54.0	45

Nature of Indigos.	Number of cubic centimeters of hypochlorite of lime to discolorize 100^{cc} of dissolution.	Indigo per cent.
CARACAS	96.0	80
Idem . . .	90.0	75
Idem . . .	78.0	65
Idem . . .	69.6	58
Idem . . .	66.0	55
GUATEMALA.		
Kurpath . . .	92.4	77
Blue . . .	81.6	68
Violaceous . .	63.6	53
MADRAS	68.4	57
Idem . . .	48.0	40
Idem . . .	38.4	32
MANILLA.		
Blue . . .	60.0	50
Dark . . .	48.0	40
Very Dark . .	46.8	39

Nature of Indigos.	Number of cubic centimeters of hypochlorite of lime to discolorize 100cc of dissolution.	Indigo per cent.
BOMBAY.		
Light Blue	42.0	35
Tarnish	36.0	30
Spotted	33.6	28
Brown-Black	32.4	27
PHILIPPINES	50.4	42
POLYG. TINCT.	51.6	43
Idem	32.4	27
Idem	16.8	14

CHAPTER XXIII.

OBSERVATIONS ON THE PRECEDING EXPERIMENTS.

By those different processes we have obtained the composition of 38 varieties of indigo, and afterwards we have judged which was the most convenient process to estimate their value in coloring principle.

1. The first method or elementary analysis is too long, and requires a great knowledge of chemical manipulations. It gives exactly the proportions of coloring matter.

2. The estimation of water and ashes is a very simple operation that any practitioner can make, and the more an indigo contains of

water and ashes, the less coloring matter it contains.

3. The process of assay by the vat, and precipitation of indigotine is very long, and does not give very exactly the proportion of coloring matter. The first way to operate gives a mean difference of 1.5 per cent.; the second 0.93, and the third from 0.3 to 0.5; then when you wish to estimate the quantity of indigotine contained in a commercial indigo, in precipitating the coloring matter from a vat; the third process is the best.

4. The process to exhaust a vat by wool to estimate the quantity of coloring matter, I do not recommend; the differences are too great, and the process too long, but it is the best method to ascertain the quality of an indigo.

5. The processes of assay by the sulphate of indigo are those I recommend. The use of dyeing can be left aside; for it is too long, and gives only a difference of 1.3 to 1.5.

6. The use of the colorimeter and hypochloride of lime does not present all the above

inconveniences; the processes are rapid, exact, and do not require much manipulation.

I recommend them particularly; for with a little practice, any practitioner can arrive in a short time to make them quickly.

In about one hour by the colorimeter, 12 or 15 assays can be made, and as much by the hypochloride of lime.

I resume below in one table, the quantity of pure indigotine found in all the varieties of indigo by the different processes.

Comparative Table of the Quantities of Indigotine found in Commercial Indigos by the different Processes.

NATURE OF THE INDIGO.	Quantity of indigotine by immediate analysis.	Estimation by Precipitation of Indigotine from the Vat.			Estimation by dyeing with the vat.	Estimation by the Sulphuric Dissolution of Indigo.		
		First process.	Second process.	Third process.		By dyeing.	By colorimeter.	By hypochlorite of lime.
JAVA.								
Surfine . . .	96.0	94.5	95.0	95.5	93.0	95.0	96.0	96.0
Purple . . .	89.0	87.0	87.7	88.9	86.0	88.0	89.0	89.0
Fine . . .	88.0	87.0	87.5	88.0	84.0	86.0	88.0	88.0
Surfine purple . .	84.0	82.0	82.6	83.8	80.0	80.0	84.0	84.0
Surfine violet . .	74.0	72.0	72.4	73.9	70.0	73.0	74.0	74.0
Fine blue . .	73.0	71.5	72.6	72.8	69.0	72.0	73.0	73.0
Fine violet . .	71.0	69.9	70.0	70.9	67.0	70.0	71.0	71.0
Fine . . .	71.0	69.9	70.0	70.9	67.0	70.0	71.0	71.0
Blue-black . .	56.0	54.0	54.9	55.8	52.0	57.0	56.0	56.0
BENGALIS.								
Surfine purple .	95.0	93.5	94.0	94.9	91.0	94.0	95.0	94.0
Fine violet . .	85.0	83.0	84.3	84.9	81.0	83.0	85.0	83.0
Surfine violet . .	82.0	80.5	84.2	81.8	79.0	72.0	74.0	73.0
Fine violet . .	74.0	72.2	72.4	73.9	72.0	74.0	75.0	75.0

Comparative Table of Quantities of Indigotine—Continued.

NATURE OF THE INDIGO.	Quantity of indigotine by immediate analysis.	Estimation by Precipitation of Indigotine from the Vat.			Estimation by dyeing with the vat.	Estimation by the Sulphuric Dissolution of Indigo.		
		First process.	Second process.	Third process.		By dyeing.	By colorimeter.	By hypochlorite of lime.
BENGALIS—*Continued.*								
Fine violet-red .	75.0	73.0	74.4	74.8	73.0	63.0	66.0	65.0
Violet . . .	66.0	64.5	64.9	65.7	62.0	43.0	45.0	45.0
Low cupreous .	45.0	44.0	44.8	45.1	41.0	80.0	81.0	80.0
CARACAS. . . .	81.0	79.0	80.8	81.0	78.0	80.0	81.0	80.0
Idem . . .	75.0	73.0	74.4	74.9	71.0	73.0	75.0	75.0
Idem . . .	66.0	64.4	64.9	65.9	62.0	64.0	66.0	65.0
Idem . . .	59.0	57.5	58.0	59.0	55.0	57.0	59.0	58.0
Idem . . .	56.0	54.0	54.9	55.9	52.0	54.0	56.0	55.0
GUATEMALA.								
Kurpath . . .	78.0	75.8	76.2	77.9	75.0	75.0	78.0	77.0
Blue . . .	68.0	65.0	66.6	67.8	63.0	65.0	68.0	68.0
Violaceous . .	54.0	52.2	53.4	54.0	51.0	50.0	54.0	53.0
MADRAS. . . .	58.0	55.4	56.6	57.9	55.0	55.0	58.0	57.0
Idem . . .	42.0	40.9	41.1	42.1	40.0	40.0	42.0	40.0
Idem . . .	32.0	30.0	30.9	31.9	30.0	30.0	32.0	32.0

Comparative Table of Quantities of Indigotine—Concluded.

NATURE OF THE INDIGO.	Quantity of indigotine by immediate analysis.	Estimation by Precipitation of Indigotine from the Vat.			Estimation by dyeing with the vat.	Estimation by the Sulphuric Dissolution of Indigo.		
		First process.	Second process.	Third process.		By dyeing.	By colorimeter.	By hypochlorite of lime.
MANILLA.								
Blue	50.0	48.7	49.2	49.8	48.0	48.0	50.0	50.0
Dark	42.0	40.9	41.2	42.0	40.0	40.0	42.0	40.0
Very dark	40.0	39.0	39.1	39.9	38.0	38.0	40.0	39.0
BOMBAY.								
Light blue	35.0	33.7	34.3	34.9	32.0	34.0	35.0	35.0
Tarnish	31.0	30.0	30.8	31.1	28.0	30.0	31.0	30.0
Spotted	29.0	27.4	27.9	28.8	25.0	28.0	29.0	28.0
Brown-black	27.0	25.2	26.4	27.1	24.0	25.0	27.0	27.0
PHILIPPINES	43.0	42.0	42.3	43.2	40.0	40.0	43.0	42.0
POLYGONUM TINCTORIUM	43.0	42.0	42.4	43.0	40.0	40.0	43.0	43.0
Idem	28.0	25.4	25.9	27.9	25.0	25.0	28.0	27.0
Idem	14.0	11.5	12.4	14.1	12.0	13.0	14.0	14.0

These different experiments were executed by the editor in the manufacture Impériale des Gobelins during the year 1850 in conjunction with other experiments on different coloring matters. What he has given here will be sufficient to enable the manufacturer of Blues and Carmine to judge of the relative value of the different indigos he uses in fabrication.

INDEX.

A

B

E

F

G

H

I

K

L

M

N

O

P

R

S

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THE INTERSECTION AND DEVELOPMENT OF SURFACES, WITH APPLICATIONS.—*The Intersection of Cylinders and Cones*, Plate XIV. *The Delineation and Development of Helices, Screws and Serpentines*, Plate XV. Application of the helix—the construction of a staircase, Plate XVI. The Intersection of surfaces—applications to stop-cocks, Plate XVII. *Rules and Practical Data*—Steam, Unity of heat, Heating surface, Calculation of the dimensions of boilers, Dimensions of firegrates, Chimneys, Safety-valves.

THE STUDY AND CONSTRUCTION OF TOOTHED GEAR.—Involute, cycloid, and epicycloid, Plates XVIII. and XIX. Involute, Fig. 1, Plate XVIII. Cycloid, Fig. 2, Plate XVIII. External epicycloid, described by a circle rolling about a fixed circle inside it, Fig. 3, Plate XIX. Internal epicycloid, Fig. 2, Plate XIX. Delineation of a rack and pinion in gear, Fig. 4, Plate XVIII. Gearing of a worm with a worm-wheel, Figs. 5 and 6, Plate XVIII. *Cylindrical or Spur Gearing*, Plate XIX. Practical delineation of a couple of Spur-wheels, Plate XX. *The Delineation and Construction of Wooden Patterns for Toothed Wheels*, Plate XXI. *Rules and Practical Data*—Toothed gearing, Angular and circumferential velocity of wheels, Dimensions of gearing, Thickness of the teeth, Pitch of the teeth, Dimensions of the web, Number and dimensions of the arms, wooden patterns.

CONTINUATION OF THE STUDY OF TOOTHED GEAR.—Design for a pair of bevel-wheels in gear, Plate XXII. Construction of wooden patterns for a pair of bevel-wheels, Plate XXIII. *Involute and Helical Teeth*, Plate XXIV. *Contrivances for obtaining Differential Movements*—The delineation of eccentrics and cams, Plate XXV. *Rules and Practical Data*—Mechanical work of effect, The simple machines, Centre of gravity, On estimating the power of prime movers, Calculation for the brake, The fall of bodies, Momentum, Central forces.

ELEMENTARY PRINCIPLES OF SHADOWS.—*Shadows of Prisms, Pyramids and Cylinders*, Plate XXVI. *Principles of Shading*, Plate XXVII. *Continuation of the Study of Shadows*, Plate XXVIII. *Tuscan Order*, Plate XXIX. *Rules and Practical Data*—Pumps, Hydrostatic principles, Forcing pumps, Lifting and forcing pumps, The Hydrostatic press, Hydrostatical calculations and data—discharge of water through different orifices, Gaging of a water-course of uniform section and fall, Velocity of the bottom of water-courses, Calculation of the discharge of water through rectangular orifices of narrow edges, Calculation of the discharge of water through overshot outlets, To determine the width of an overshot outlet, To determine the depth of the outlet, Outlet with a spout or duct.

APPLICATION OF SHADOWS TO TOOTHED GEAR, Plate XXX. *Application of Shadows to Screws*, Plate XXXI. *Application of Shadows to a Boiler and its Furnace*, Plate XXXII. *Shading in Black—Shading in Colors*, Plate XXXIII.

THE CUTTING AND SHAPING OF MASONRY, Plate XXXIV. *Rules and Practical Data*—Hydraulic motors, Undershot water wheels, with plane floats and a circular channel, Width, Diameter, Velocity, Number and capacity of the buckets, Useful effect of the water wheel, Overshot water wheels, Water wheels with radial floats, Water wheel with curved buckets, Turbines. *Remarks on Machine Tools*.

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OBLIQUE PROJECTIONS.—Application of rules to the delineation of an oscillating cylinder, Plate XLI.

PARALLEL PERSPECTIVE.—Principles and applications, Plate XLII.

TRUE PERSPECTIVE.—Elementary principles, Plate XLIII. Applications—flour mill driven by belts, Plates XLIV. and XLV. Description of the mill, Representation of the mill in perspective, Notes of recent improvements in flour mills, Schiele's mill, Mullin's "ring millstone," Barnett's millstone, Hastie's arrangement for driving mills, Currie's improvements in millstones; *Rules and Practical Data*—Work performed by various machines, Flour mills, Saw mills, Veneer-sawing machines, Circular saws.

EXAMPLES OF FINISHED DRAWINGS OF MACHINERY.—Plate A, Balance water-meter; Plate B, Engineer's shaping machine; Plate C D E, Express locomotive engine; Plate F., Wood planing machine; Plate G, Washing machine for piece goods; Plate H, power loom; Plate I, Duplex steam boiler; Plate J, Direct-acting marine engines.

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