An experimental examination of the last edition of the Pharmacopoeia Londinensis; with remarks on Dr. Powell's translation and annotations / [Richard Phillips].

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# EXPERIMENTAL EXAMINATION

OF THE

LAST EDITION

OF THE

# Pharmacopoeia Londinensis:

WITH REMARKS

ON

DR. POWELL'S TRANSLATION

AND

ANNOTATIONS.

BY RICHARD PHILLIPS.

LONDON:

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# ADVERTISEMENT.

Many of the experiments recorded in the following pages have been detailed in the tenth number of the London Medical Review; accompanied in conformity with the nature of that work, by a critical examination of the Pharmacopæia and of Dr. Powell's translation. The whole of these experiments I performed, and furnished most of the critical remarks.

Several medical friends have advised me to republish the detail and the results of these experiments, not only for the purpose of circulating them more widely, but also to obtain an opportunity of relating experimental minutiæ, which the limited space afforded by the Review necessarily excluded.

I am now able to add the results of much subsequent investigation; and on this occasion I have not introduced that part which is merely critical: this I think can scarcely be requisite, because those peculiarities which occur so frequently in the translation, as well as those from which indeed the original is not altogether exempt, are sufficiently conspicuous to insure observation.

I have confined my attention as much as possible, to such points as my avocation and pursuits have brought more immediately before me; and to those erroneous opinions and mis-statements of facts which are likely to prove inconvenient to the operator, and dangerous to the community. Whoever has examined the Pharmacopæia and the official Translation can, I am persuaded, entertain no doubt of the necessity of the undertaking; for although individual error may be insignificant, yet when established and enforced by the authority of law, it becomes truly formidable.

In the course of this investigation it will appear that I have had access to the "Specimen Pharmacopæiæ," sent by the College of Physicians to those persons who appeared likely to afford assistance in effecting the projected reformation: it may therefore be inquired why I did not submit my remarks and experiments to the consideration of the College, previously to the completion of the Pharmacopœia; instead of publishing them after its appearance, when the probability of their being useful was diminished? In reply to this very natural inquiry it will be sufficient to state, that I saw the specimen at first merely by accident, in the possession of a medical acquaintance who had received it officially; and having obtained an opportunity of perusing it, I was satisfied that I could render no essential service by remarks upon an accumulation of imaginary improvements; for this performance, whilst it evinced no want of speculation, demonstrated great deficiency of experimental research.

My usual occupations prevented me from submitting to trial many of the proposed alterations, which in a theoretical point of view appeared of very questionable propriety, until after the circulation of the "Specimen alterum." This I had also an opportunity of inspecting; and having learned, that although uninvited by the College, there would be no impropriety in addressing them on the subject of the intended work, I prepared some observations and sent them, with an offer of communicating the results of those experiments which I intended to perform for ascertaining several doubtful points, provided the College were disposed to receive them. Here the matter rested. But had I seen any reason to suspect the existence of the means which it since appears were adopted for conducting the requisite experiments, the fear of being thought presumptuous would probably have deterred me from tendering my services.

The latter part of the following quotation from Dr. Powell's preface to his translation, appears to be intended to show that a powerful union of talent and opportunity was formed for the purpose of experimental investigation. "Nor did they, [the committee of fellows] in their progress, overlook, but, on the contrary, derived great assistance from the recent Pharmacopæiæ, edited by the Colleges of Edinburgh and Dublin. They also established, as had been done in a former instance, a most important intercourse with the Society of Apothecaries, who appointed a committee for the purpose of co-operating with the College, in the use of their extensive laboratory, and in bringing to the test of that sort of experiment

upon a large scale, which could alone render the suggestions of science practically useful, the several processes which were communicated by the College."

Although much obligation to the Dublin and Edinburgh Pharmacopæias is here avowed, it must nevertheless be inferred from this quotation that a considerable number of the processes now directed had actually originated with the College; that all had been submitted to the experimental examination of the committee of the Apothecaries' company, and that they were finally adopted, not merely in consequence of their being practicable, but on account of their tried excellence. I have however shown that almost every change which can be considered as an improvement, as well as some alterations which are the reverse, have been copied from or at least suggested by the Dublin and Edinburgh Pharmacopæias, or by Dr. Duncan's Dispensatory. It is by no means requisite that to be eligible a process should be original, and as it was unquestionably expedient for the College freely to borrow from every quarter, it was surely no less expedient to ascertain that the loan was worthy of acceptance. Such however is the quality of some of the alterations, that to suppose them untried is to adopt the only creditable apology for their appearance in the performance, which I have now freely, but I trust, candidly examined.

After a part of this work had been printed I received from Dr. Bostock his "Remarks on the Nomenclature of the New London Pharmacopecia," and it was at first observations upon the chemical names adopted by the College; but I soon found that to repeat every just reflection would be to copy almost the whole, and I have therefore preferred giving this general statement of my coincidence with the sentiments of that acute and scientific essay.

Presuming that the College in conducting the reformation of their late labours, so imperiously and I may add so universally demanded, will be anxious to substitute actual for conjectural improvement, I now repeat the offer of my services, should it appear from the contents of the following pages that I could forward in the slightest degree, this necessary and arduous undertaking.

R.P.

29, Poultry, 1st September, 1811. ( vii. )

P. P.

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10 September, 1811.

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# EXPERIMENTAL EXAMINATION, &c.

BEFORE I proceed to the principal object of this Essay, I shall make a few observations upon the alteration of the names of measures. This was not only unnecessary, but by the mode in which it has been carried into effect has been productive of much ambiguity and some absurdity. It is at first laid down as a general rule, that all fluids are to be measured unless specially directed to be weighed; this rule is then rendered absolutely nugatory, for in order to insure the measurement of fluids, and to prevent the ambiguity which arose from employing the same words to designate pound and pint with their subdivisions, the following terms are introduced to express measured quantities only: of these it will be observed two are original, and the others modifications of terms previously employed to describe certain portions of solids.

Octarius Fluiduncia Fluidrachma Minimum Pint
Fluidounce
Fluidrachm
Minim, or 60th part
of a fluidrachm

After this plan had been established, it would not be expected that any other meaning than pound could be attached to libra, yet the College seem to suppose that when employed to express a quantity of fluid, it may retain its former meaning of pint: accordingly on the first occasion for directing a fluid to be weighed, libra is preceded by pondere, an addition, which however inconsistent, must be considered as essential to the directions for estimating a fluid by weight, and therefore to be employed and expected on all similar occasions.

It will be difficult to select an instance more illustrative of the confusion which these alterations have occasioned, than one which occurs in the fourth process, in the directions for preparing

### ACIDUM MURIATICUM.

R

Sodæ muriatis exsiccatæ libras duas, Acidi Sulphurici pondere libram cum semisse, Aquæ distillatæ octarium cum semisse;

Acidum cum aquæ selibra &c.

Here, as well as in several other cases which shall be pointed out, the quantity of fluid intended to be expressed by selibra is not ascertainable; for when directing the sulphuric acid to be weighed in this very process, libra is preceded by pondere, and as the same mode of expression is not afterwards employed, selibra cannot refer either to weight or measure: if however selibra actually mean half a pint, as by Dr. Powell it is rendered, the College must have forgotten their recently proposed term octarius, used immediately before in the same process, to designate a quantity of the same fluid.

In the directions for preparing those compounds of mercury occupying the place to which, according to the arrangement adopted, they properly belong, this fluid metal is invariably directed to be weighed; libra being, as in the case of the sulphuric acid just noticed, preceded by pondere; it must therefore be considered as subject to the general rule directing the measurement of fluids, although it is in no one instance clearly directed to be measured. In the following preparations of mercury not arranged with Præparata ex Hydrargyro, viz. Pilulæ Hydrargyri, Emplastrum Ammoniaci cum Hydrargyro, Emplastrum Hydrargyri, Unguentum Hydrargyri fortius, Unguentum Hydrargyri Nitratis, the word libra is not accompanied by pondere, nor yet uncia or drachma by fluid; it was therefore questionable whether the mercury is to be weighed or measured; this difficulty was however removed by the fortunate expedient of referring to the "Tabula," and from the quantity of mercury which a given portion of the Pilulæ Hydrargyri is intended to contain, it appeared that the mercury in this case, and probably in all the remaining doubtful cases, is to be weighed.

The following are the instances, in addition to that already noticed, in which the quantity of fluid intended to be employed is undiscoverable, because not expressed according to either of the methods previously adopted.

SODÆ SUBCARBONAS.

..... Hæc vaporet ad libras duas, .....

LIQUOR CUPRI AMMONIATI.

..... Aquæ distillatæ libram; .....

LIQUOR PLUMBI ACETATIS.

..... decoque ad libras sex, ......

A 2

# LIQUOR PLUMBI ACETATIS DILUTUS. ..... Liquoris Plumbi acetatis drachmam, ..... EXTRACTUM ANTHEMIDIS. ..... Decoque ad libras quatuor, ...... EXTRACTUM COLOCYNTHIDIS. ..... Decoque ad libras quatuor, ..... OXYMEL. ..... Acidi acetici libram; ..... OXYMEL SCILLÆ. ..... Aceti Scillæ libras duas; ..... SYRUPUS PAPAVERIS. ..... decoque ad libras duas, ..... SYRUPUS RHAMNI. ..... Succi defæcati libræ ..... SYRUPUS ROSE. ..... consume ad libras duas cum semisse: .....

Having now shewn how imperfectly these changes have been introduced, I shall state why I think them unnecessary. It appears to have been well understood that libra used to signify a quantity of fluid, meant a pint, the ambiguity occasionally attendant upon it arose entirely from its too limited and varying use; the remedy for this defect would have been a simple declaration that libra applied to all fluids (mercury excepted) denoted a pint, consisting of sixteen ounces of eight drachms each, and each drachm of 60 parts of one grain each. This rule might have been adopted without a single exception, for on examining the Pharmacopæia it will be found that every fluid employed in it, except mercury, is occasionally directed to be measured.

There can be no doubt that all fluids, and especially the more dense, are apportioned with greater precision by weighing than by measuring, and as might have been expected, nearly all the exceptions to the general method of measurement, occur in some instances of the employment of mercury, and the stronger acids: now although the measuring of acids appears to me to be in all cases sufficiently accurate for the purposes of medicine, yet two methods of effecting the same purpose being adopted by the College, it might reasonably be supposed that the more exact one had been resorted to in those preparations in which accuracy is of the greatest importance; the reverse is however unfortunately the case, as may be proved by evidence which, although others may mistrust, the College cannot object to. The following are the preparations in which the acids are to be weighed.

Now upon examining these preparations, it will appear that the last only would be essentially altered, even by a much greater variation in the proportion of the acid than could arise from the difference of the two methods in question; it will also be found that the acids employed, either enter into combination, or are partially or totally decomposed; and these circumstances, as they diminish

or destroy the power of the acid, render accurate adjustment of less consequence.

To render incontrovertible what I have advanced on this head, I subjoin the names of those preparations in which the acids are to be measured: among these are included those in which the acids employed are exhibited internally, unaltered, and uncombined.

Acidum Benzoicum

— Citricum

— Sulphuricum dilutum

— Nitricum dilutum

Liquor Ferri alcalini

Tinctura Ferri muriatis

Argenti Nitras

Infusum Rosæ

Unguentum Hydrargyri nitratis

I shall now proceed to state the results of the various experiments instituted for the purpose of clearing up many doubts and difficulties in the processes proposed in the new Pharmacopæia. It can hardly be expected that minute attention should be given to every part of so multifarious a production; I have accordingly selected such parts only as seemed to present the strongest claims to investigation.

#### ACIDUM ACETICUM.

The acid which distilled vinegar contains, is undoubtedly the acetic; but before the name of one substance is affixed to any other, it should be ascertained that they produce exactly the same effects under circumstances precisely similar: it is not sufficient that the general results of their action resemble each other. Examining

upon this principle the name now adopted for distilled vinegar, it appears to be incorrect. When a neutral solution of acetate of potash is boiled, it remains colourless, and the salt obtained by evaporating it to dryness is white, provided the acid has been procured by decomposing an acetate; but when distilled vinegar is thus employed, the solution quickly exhibits a deep reddish brown colour, and during evaporation a considerable quantity of carbonaceous matter is deposited, in consequence of the action of the salt upon the mucilage contained in the vinegar. This incorrect name is also inapplicable, for one class of medicines consists of compounds retaining the former appellation acetum, although prepared with what is now termed acidum aceticum.

In the remarks upon acetate of potash, Dr. Powell seems to imagine, that this carbonaceous deposit is derived from the decomposition of the acetic acid: but that it has a different origin is demonstrated not only by the fact just mentioned, but also by this circumstance; when carbon has been deposited, the solution remains neutral instead of being alcaline, as must be the case if a portion of acid had been decomposed.

The process now proposed for obtaining this acid is unnecessarily expensive. The first eighth part yielded by distillation is to be rejected. The best malt vinegar, unadulterated by sulphuric acid or colouring, has a sp. gr. of 1.0204, and the first eighth obtained by distillation from vinegar of this strength is of sp. gr. .99712; it probably contains a portion of alcohol rendering it lighter than water; its taste is distinctly acid, and a fluidounce decomposes from 4.5 to 5 grains of precipitated carbonate of lime; the subsequent six-eighths are of the sp. gr. 1.0023, and a fluidounce decomposes 8.12 grains of pre-

cipitated carbonate of lime; a similar quantity of sp. grav.
1.007 decomposes from 15 to 16 grains of precipitated
carbonate of lime, or 13.8 grains of marble: from this
statement it appears, that when vinegar of the sp. gr.
1.0204 is distilled, about one twelfth of the acid obtained
exists in the first eighth which passes over. It is certainly
improvident to reject so large a portion of the product
on account of its dilution; especially as there is no instance of the employment of this acid, at least internally,
without previous evaporation of a portion of the water,
or subsequent dilution of the compounds of which it
forms a part; these circumstances shewing, that it is not
requisite to procure the acid, either of very equal or very
great strength.

In the first edition of the translation, the sp. gr. of distilled vinegar is stated to vary from 1.007 to 1009: in the long, but inaccurate and incomplete list of errata since printed, 1.095 is directed to be substituted for 1.009, without any alteration of 1.007. I have certainly never found distilled vinegar of a sp. gr. so high as 1.009, yet it probably has occurred of this strength; but 1.095 is greater than the sp. gr. of undistilled vinegar, and I have already shewn that by the abstraction of the colouring matter and other impurities, and on account of the great portion of acid remaining with them in the retort after distillation, its sp. gr. is reduced from 1.0204, to rather less than 1.0023: the substitution therefore, of 1.095 for 1.009 must be considered an error.

In the second edition the sp. gr. is omitted, and instead of this method of ascertaining the strength of distilled vinegar, the quantity of marble that a portion of it is capable of decomposing is inserted: this however, is incorrectly given, for as already mentioned, a fluidounce will decompose 13.8 grains instead of about 10 as stated by Dr. Powell: the acid which I employed for this purpose was of sp. gr. 1.007 the weaker mentioned in the first edition of the translation. The error in this instance, as well as in another which I shall presently notice, probably arose from employing a greater "lump" of marble than the acid could decompose, and insufficiently drying the residuum; the moisture being weighed with the marble, and consequently estimated as marble undecomposed.

The alteration in this process seems to have been suggested by the Edinburgh and Dublin Pharmacopæias; the former directing the rejection of one third and the latter one sixth of the vinegar distilled.

I have not been able to procure this acid from Apothe-caries' Hall. The distilled vinegar procured there is of sp. gr. 1.007; this it will be observed, is considerably stronger than the product which I obtained by following the directions of the College for preparing Acidum aceticum.

## ACIDUM BENZÖICUM.

This very inactive medicine appears to have been retained without sufficient reason; it constitutes a part of one preparation only, the Tinctura Camphoræ composita, and as in this there is an equal quantity of opium, it is evident that the exhibition of the tincture must be so regulated as not exceed the dose of the more powerful ingredient. Now by referring to Dr. Powell's table of doses it appears, that of benzoic acid, six times as much may be given as of opium, so that in the only preparation into which benzoic acid enters, one sixth of a dose only can be exhibited.

Scheele's process for obtaining this acid is now adopted instead of Chaptal's: an alteration which cannot be regarded as an amendment; the present operation is not only much more tedious and expensive, but from Mr. Brande's experiments it appears that the quantity obtained is less in the proportion of about 45 to 48, and the only difference in the products is, that the acid procured by Chaptal's method is slightly impregnated with oil; but this cannot be considered of importance, for the quantity of this acid which the largest dose of the Tinctura Camphoræ composita contains, scarcely amounts to one grain.

This acid procured at Apothecaries' Hall, possesses the peculiar odour of that obtained by the former method.

#### ACIDUM MURIATICUM.

Premising that the cost of muriate of soda and sulphuric acid is nearly equal, I shall proceed to state the results of experiments, instituted for the purpose of comparing the former and present process for obtaining this acid.

	Muriate Soda.	Solph. Acid.	Water.	Cost.	Pro luct.	Sp. gr.	Marble -
Process of 1787.	35.	21.	17,5	56.	29.75	1.188	15.09
Process of 1809.	32.	24.	39.4	56.	43.68	1.142	17.16
Process of 1787 modified.	35.	21.	22.0	56.	35.00	1 174	16.43

Excepting the cost and sprecific gravity, the figures represent parts by weight.

By comparing these statements, it will appear that the present process at an equal expence, affords a greater quantity of muriatic acid than the former, in the proportion of about 17 to 15, and cæteris paribus, this might be considered as the relative value; but the advantage

obtained is counterbalanced by the great increase of water with which the acid is diluted, a solvent power of 17 being contained in nearly 44 parts, whereas an equal solvent power by the former method would be contained in about 34 parts. By this excessive dilution, the muriatic acid of the new Pharmacopæia is reduced from the intended sp. gr. of 1.17 to 1.142: acid of this sp. gr. is undoubtedly sufficiently concentrated for any medicinal purpose, if it usually had been, or was now intended to be employed of this strength; the Collegiate experimentalist must indeed have procured acid by some other method, the sp. gravity of which was about 1.17, for it is nearly correctly stated, that a fluidounce, of this strength, is capable of decomposing 240 grains of marble; but a fluidounce of muriatic acid, obtained by the present process, decomposes only about 204 grains of marble, instead of 240.

It seemed probable that an additional quantity of water might be advantageously employed to condense the vapour which escaped during the former process, and this I found was the case, as appears on comparing the result of the modified process with the former, the addition of water having increased the solvent power from 15.09 to 16.43.

Dr. Powell has stated that three parts of sulphuric acid are required to decompose four parts of muriate of soda: these proportions are now adopted by the College, and being recommended by Vauquelin, I should scarcely have questioned their accuracy, if the results of repeated experiments had not indicated the use of a smaller proportion of acid. It will be seen in the experiments above stated, that 21 parts of sulphuric acid and 35 of muriate of soda, yielded muriatic acid possessing a solvent power of 16.43; whilst with 24 parts of sulphuric acid, and 32

of muriate of soda, a solvent power of 17.16 only was procured: but if the solvent power had been in proportion to the quantity of sulphuric acid employed, a power of 18.76 ought to have been obtained: for 21:16.43::24 :18.76. Then as solvent power 16.43 is produced by 21 of sulphuric acid, 17.16 the greatest power procurable from 32 of muriate of soda, requires only 21.9 instead of 24, as now employed.

In the late Pharmacopæia, the acid and water previously mixed, were directed to be added to the salt in the retort: this method is now reversed, and the reason which Dr. Powell has assigned for the change is, to avoid endangering the vessel. The former mode is however much more convenient, and having uniformly employed it with safety, I cannot conjecture from what imaginary cause it has been supposed dangerous.

It is certainly an improvement to put a part of the water employed, into the receiver as now directed, instead of mixing the whole of it with the sulphuric acid and muriate of soda: it has been long practised by most operators, but the College probably derived it from the Edinburgh or Dublin Pharmacopæia.

Muriatic acid, obtained from Apothecaries' Hall, is of sp. gr. 1.158, which is nearly intermediate between that directed by the College, and that afforded by the present process.

#### ACIDUM NITRICUM.

The proportion of sulphuric acid formerly employed to decompose nitre for the preparation of this acid, is now considerably increased, in order, as stated by Dr. Powell, to obtain it free from nitric oxide. It is also remarked, that the acid procured by the new process, is "greater in value than where half the weight only of sulphuric

acid is employed; " and the following statement of experiments, presented to the committee, is inserted in the translation, to show the propriety of the alteration to which this process has been subjected.

" Drie		Colour of Acid obt.	Sp. gr.	Weight of Product,	Marble dissblved.	Relative value.
6	3	Red	1,530	- 3	70	21
6	6	White	1,500	4	73	29
Present 60	29	Red	1,456	30+	62	19+"

It happens most unfortunately, that every intelligible part of this statement demonstrates the reverse of what it was adduced to prove. I shall presently show, that the only approximation to a fair deduction which it offers, is in proof of the impropriety of the alteration, and consequently that the former process is preferable to the present.

The Collegiate experimentalist has strangely omitted to insert the expence at which these products were severally obtained: this I shall supply, in order to ascertain their relative values, which can only be found by comparing their cost and strength; that preparation certainly being of the greatest value, which at a given expence, possesses most solvent power.

Nitre is of about three times the value of sulphuric acid: and estimating the cost of the ingredients of the first experiment to be 84, that of the second will be 96; the first product being 3 parts each decomposing 70 parts of marble, and the second product 4 parts each decomposing 73 parts of marble. Cost 84 therefore decomposes 210, and cost 96 decomposes 292 parts. If then with an expense of 84 we acquire a product of 210, the expense of 96 should produce 240, to be of equal value; but we

find that 96 produces 292, and is therefore of greater relative value in the proportion of 29.2 to 24, or of 25.5 to 21, instead of 29 to 21, as stated in this table of experiments: and these being the proportions of marble dissolved, it is evident that the calculation has been made upon the very incorrect assumption, that greater power, although obtained at an increased expense, necessarily

augments the relative value of the product.

When 30 parts of sulphuric acid are employed with 60 of nitre, it is stated that the sulphuric acid is not redundant: we are not however informed whether any nitre remains undecomposed, but may fairly infer that this is the case, from the College having now directed the quantity of acid to be doubled; very unnecessarily indeed, if any dependence is to be placed on the foregoing table. We are there told, that 30 parts of sulphuric acid, with 60 of nitre, produce 30 parts of nitric acid, each dissolving 170 of marble, = 21.0; whilst 29 parts of sulphuric acid with 60 of nitre, produce 30 parts of nitric acid, each dissolving .62 of marble, = 18.6: the aiminution of one part in thirty of sulphuric acid diminishing the solvent power of the nitric acid produced, no less than 24 parts in 210. Now 60 parts of sulphuric acid, with an equal quantity of nitre, are stated to produce 40 parts, each dissolving .73, = 29.2; giving for the addition of 30 parts of acid an increased solvent power of only 82 parts in 210, an increase which would be afforded by the addition of less than four parts of sulphuric acid; for 24:1::82: 3.41.

I have given these calculations, as the results which ought to have influenced the College, if credit had been given to the statements upon which they are grounded; but on repeating some of these experiments, I find that

little reliance is to be placed upon their accuracy; one assertion is indeed so palpably erroneous, that it carries with it its own refutation. One hundred parts of the product by the first experiment, of sp. gr. 1.53, dissolve, it is stated, 70 parts of marble; whereas 100 parts of the second, of sp. gr. 1.50, dissolve 73 parts: acid of inferior density being thus stated to possess superior power. The first process I have not repeated, it differs but little from the third, the product of which is not red, nor is that of the second white; they are both of a pale greenish yellow colour. The sp. gr. of the second is nearly correct, but that of the third is 1.51, instead of 1.456, as expressed in the table. From what I have already mentioned it must be evident, that the quantity of marble dissolved by 100 parts of the first or second product, is incorrectly stated.

It appears therefore that no advantage results from the new process, in an economical point of view, and as the product obtained by it is not less coloured than that procured by the former, it cannot in any sense be considered as "better nitric acid."

It has seemed so desirable to the College to obtain colourless nitric acid, that with this intention they have not only directed twice the former quantity of sulphuric acid employed for the decomposition of the nitre, but according to Dr. Powell, they have also ordered that the distillation should be continued, only, "until a red vapour arises,"—"donec vapor ruber prodeat." Now if the English were actually a translation from the Latin, instead of the reverse, it might be imagined that donec had been improperly rendered by "until," instead of, "as long as," for I have never yet prepared this acid without the occurrence of red vapour, immediately upon the

application of heat to the retort, and its continuance until the operation was nearly finished; so that if the directions contained in Dr. Powell's work had been observed, I never should have procured any nitric acid.

Although the College have not succeeded in their intention of procuring colourless nitric acid; their failure, in this instance at least, is unimportant, for no advantage could have arisen from success. When nitric acid of the deepest red colour is considerably diluted, as must be the case previous to exhibiting it, the water so completely expels the nitric oxide from which the colour is derived, that no difference is discoverable, between the products obtained by diluting red and colourless nitric acid.

The College seem to have been aware that they have directed the employment of an unnecessarily large quantity of sulphuric acid; and apprehending that some part of the redundancy may rise with and contaminate the product, they have ordered it to be re-distilled with a small portion of nitre, without directing any previous examination of the nitric acid, to prove the necessity of submitting it to this operation, and without considering that if such a portion only of sulphuric acid pass over as is sufficient to decompose the quantity of nitre directed, that this process might be avoided by employing a greater quantity of nitre in the first instance.

If however Dr. Powell's "additional information" may be considered authentic, the operator may presume that he is at liberty to omit the re-distillation, if he find it unnecessary; and he is directed to employ nitrate of silver, for the purpose of ascertaining that no sulphuric acid is contained in the nitric. This reagent is however incapable of effecting the end proposed, for the sulphate

of silver which might be formed, although a salt of little solubility in water, is readily dissolved by nitric acid: it is scarcely requisite to add, that nitrate of barytes should be employed for the detection of sulphuric acid; and with it I have never discovered any in the nitric acid prepared according to the direction of the College.

Nitric acid is very commonly rendered impure by muriatic acid, derived from decomposition of the muriate of soda which is usually mixed with the nitre; but no direction is given for removing this acid.

Although it cannot be reckoned very important that the acids, when intended merely for internal use, should be perfectly pure, yet for this purpose muriatic acid is at least as objectionable in admixture with nitric, as sulphuric acid is, and chemically considered it is much more so; for in preparing the nitrate of silver it will occasion considerable loss, by combining with and precipitating the oxide from its solution in the nitric acid.

In addition to the usual accurate mode of determining the strength of an acid by taking its specific gravity, the College have now introduced the very troublesome and uncertain method of ascertaining its solvent power. No other proof of the insufficiency of this method need be adduced, than that furnished by comparing the statements of the solvent power of nitric acid, given in the original, and in the translation. In the Pharmacopæia it is directed, that a fluidounce (incorrectly rendered ounce by Dr. Powell) of nitric acid of sp. gr. 1.50, should dissolve 420 grains of limestone; whilst in the experiments mentioned in page 67 of the translation, 100 parts of nitric acid of this sp. gr. dissolve 73 parts of marble; consequently 6813 grains, the weight of a fluidounce, ought to decompose 497 grains. In further illustration of the uncertainty

of this method I have to remark, that both the above statements are incorrect; for I find that this quantity of acid of sp. gr. 1.50, decomposes 476 grains of marble; greater than the quantity in the original by 56 grains, and less than that of the translation by 21 grains.

If however, this method be persisted in, the quantity of acid should be determined by weighing, instead of measuring, as now directed; for it is evident that a variation of one minim in the measure, must occasion a difference of nearly one grain in the result.

In the translation it is said, that the present process was adopted in consequence of the experiments therein related: i. e. in consequence as has been shown, of erroneous inferences from incorrect statements: but as it is also mentioned that "Frederick Hoffman used equal parts of the salt and acid," it may perhaps be safely concluded, that ancient authority has been preferred to modern experiment.

The nitric acid procured at Apothecaries' Hall, is of a greenish yellow colour, and of the sp. gr. 1.424.

#### ACIDUM NITRICUM DILUTUM.

In the observations annexed to this article in the translation, it is asserted, that "one ounce of nitric acid by measure, is equal to about two ounces by weight." The occurrence of an error so extravagant, can be accounted for only by supposing that sulphuric acid has been mistaken for nitric; not indeed that even then the statement would be exact, but it would scarcely have excited observation, and would certainly have escaped comment, when appearing in a work of such general and gross inaccuracy. The plausibility of the cause here assigned for this misstatement, is shewn by Dr. Powell's observations upon the succeeding preparation: but in whatever way it may have originated, it seems not to have been sufficiently conspicuous to obtain insertion in the list of errata of the first edition, or to escape repetition in the second.

A fluidounce of nitrie acid weighs less than one ounce and a half; for one pint of water at a medium temperature, weighs 7272 grains; a fluidounce therefore weighs 454½ grains: and as any fluid the sp. gr. of which is 1.50, is precisely one and a half time as heavy as water, a fluidounce of nitric acid must weigh 681¾ grains.

The next observation of the annotator requiring notice is, that "one ounce of this diluted acid will saturate one hundred of white marble." By the method adopted in the second edition for the correction of this mis-statement, it may be supposed to have arisen from the accidental omission of the word "grains." Although the error is diminished by the insertion of this word in the second edition, yet a very trifling calculation would have shown that it is still left very far from the truth. To have rectified this blunder as perfectly as previous error would admit, it was required merely to divide 420 by 10, for a fluidounce of diluted nitric acid contains one tenth of a fluidounce of nitric acid, which had just before been stated, though incorrectly, to be capable of dissolving 420 grains of limestone.

This short note, which consists scarcely of seven lines, contains yet another error, not indeed so vast in numbers as the former, but likely to produce greater evil in practice.

In the Pharmacopæia of 1787, the Acidum Nitricum dilutum was prepared by mixing equal weights of acid and water: in the present, nine measures of water are added to one measure of acid, and the compara-

tive strength of these mixtures is stated by Dr. Powell to be as 16 to 10. I shall examine this conclusion.

The bulk of equal weights of fluids being inversely as their specific gravity, that of the nitric acid formerly ordered is 1.0, and that of the water 1.5; together 2.5 for the bulk of mixture containing one part of acid. But in the new mode 10 parts of the mixture contain also 1 part of acid; wherefore the strength of the old preparation is to that of the new as 10 to 2.5; or as 40 to 40, instead of 16 to 10.

From a cause which cannot be mistaken, no reason whatever has been assigned for the change introduced in this preparation.

The sp. gr. of the Acidum Nitricum dilutum, is about 1.08.

#### ACIDUM SULPHURICUM DILUTUM.

I have observed in the remarks upon the last article, that Dr. Powell seems to have mistaken sulphuric acid for nitric, and on the present occasion the error has been very naturally reversed, by mistaking nitric acid for sulphuric: for what is falsely stated of one is as nearly true of the other, as from causes already mentioned might be reasonably expected.

It is now asserted that one fluidounce of sulphuric acid of the sp. gr. 1.85, weighs 11 dr. 1 scr. which wants only 1\frac{3}{4} grains of the weight of a fluidounce of nitric acid, and this error, like the former, occurs in the second edition of the translation: the fact however is that one fluidounce of sulphuric acid weighs a fraction of a grain more than 14 drachms.

In the former Pharmacopæia one part of sulphuric

both by weight: the College now order fl. oz. 1½ of the acid to be mixed with fl. oz. 14½ of water, and we are rather whimsically informed, that this change has been effected because "this mixture will be more conveniently made, and its dose more easily apportioned than the former one." Now to find the proportion of the weights of the ingredients was before sufficiently easy; to ascertain the volume of each required a little calculation, which is now requisite if the relative weights of the ingredients be sought; and the relative volumes of acid and water selected for the reasons above quoted, are in the inconvenient proportion of 3 to 29.

I shall proceed to examine the assertion, that this preparation bears to the former one the proportion of 139 to 100. The bulk of one part by weight of sulphuric acid being called 1.0, that of 8 parts of water is 148, making together 15.8, for the bulk of the mixture containing one part of acid; 47.4 of the mixture then hold 3 parts of acid, the quantity contained in 32 parts of the new preparation; and the strength of the new is to that of the old as 47.4 to 32, or as 148 to 100.

I find by the table of doses affixed by Dr. Latham to the translation of the former Pharmacopæia, that the sulphuric and nitric acids were so diluted that they might be exhibited in similar quantities with the muriatic acid, and this must certainly be considered as affording some facility to practitioners.

I have endeavoured to discover whether the medicinal power of these acids was dependant upon their chemical or physical properties, and whether their doses were established in consequence of po sessing similar power of saturation, or similar specific gravity. It is indeed evident that no very certain inferences can be derived from their action upon any particular substance, on account of their varying affinities. I shall however state the specific gravity of the former diluted acids, and that which the muriatic was directed to possess, but from which it slightly differed; and also their comparative powers of combining with soda, as nearly as a trifling variation from the specific gravity, which the undiluted acids are ordered to have, would allow:

THE PERSON OF THE PERSON	8p. Gr.	So	lver	nt Power.
Acidum Vitriolicum dilutum,	1.07.	-	-	33
Nitrosum dilutum,	1.30.	-	-	110
Muriaticum,	1.17.	-	-	140

Admitting that Dr. Latham has stated the usual and proper doses of the muriatic, and the diluted sulphuric and nitric acids, it is evident from this statement that their medicinal efficacy is intirely independant of their respective specific gravity, or saturating power, as far at least as regards soda; and it would perhaps be difficult to select any substance for which their affinity is more nearly equal, although confessedly dissimilar.

Fortunately for both practitioners and patients, the absolute medicinal virtue of the different acids, seems to have varied with the modes of their preparation; for Dr. Powell assuming on this occasion as well as on most others, that drops and minims are equal, has converted the 40 drops of the muriatic and diluted sulphuric and nitric acids given by Dr. Latham, into 40 minims, notwithstanding that the present diluted sulphuric acid is stronger than the former, in the proportion of nearly 3 to 2, and the diluted nitric acid weaker, in the proportion of 1 to 4.

Provided the more powerful acids be sufficiently diluted

to prevent them from effecting any chemical change upon the substances with which they are intended to be mixed for medicinal use, the degree of dilution cannot be considered as of much importance; the only remaining object being to facilitate their division with accuracy. If however no diluted acids of determinate strength had hitherto been employed, it would be easy to point out some circumstances to which it might be advantageous to attend in fixing the proportion of dilution; but as acids of certain strengths have been long in use, and as every practitioner is necessarily acquainted with their respective doses, the alterations effected in this department, must be considered as frivolous, as they are inconvenient and vexatious. If indeed the College were desirous in these instances of following a general rule, which on other occasions they have unceremoniously broken, it would have been easy to have converted the acids and water before ordered by weight, to measure, and this change might have been made, unattended by the serious inconveniences which now occur.

In common as I apprehend with most persons concerned in dispensing medicines, I have experienced some difficulty in determining the safest method to adopt with regard to the diluted acids; for having shown that Dr. Powell, one of the committee employed in framing the new Pharmacopæia, was unacquainted with the extent of the alterations introduced, it is evident that practitioners relying upon the statements contained in the translation, may be seriously disappointed by these articles. For the present I have judged it to be safest to keep acids diluted in both proportions, and to employ them according to the terms by which they are prescribed; presuming as is obvious, that every practitioner who uses the new terms,

is aware in what degree the new preparation differs from the former: a conclusion which may not in every instance be true.

There is one preparation in which this precaution is unavailing; the Infusum Rosæ, without having suffered any change of name, has been very considerably altered in strength, as I shall hereafter show.

The sp. gr. of the Acidum Sulphuricum dilutum is very nearly 1.11.

In the translation it is asserted, that sulphuric acid diluted with one-third of its weight of water, ceases to give out heat on its further addition; I found however that when one ounce of water had been mixed with three of sulphuric acid, and the mixture suffered to acquire the temperature of the atmosphere, about half an ounce of water at 51° immediately raised the temperature to 86°, half an ounce more to 96°, and a similar quantity to 104°.

#### AMMONIÆ CARBONAS.

Two parts of the carbonate of lime are directed, as in the former Pharmacopæia, to be employed for the decomposition of one part of muriate of ammonia. On considering the properties of these salts, it appeared to me probable that the carbonate of lime is greatly in excess; and I was convinced that this is actually the case by calculations grounded upon data not absolutely exact, which had been established for other purposes.

In order to arrive at direct experimental decision, I dissolved 100 grains of muriate of ammonia in distilled water, and added more lime to the solution than was sufficient to decompose it: having boiled the mixture till the evolu-

lution of ammonia ceased, I filtered the solution, and added water to the undissolved residuum as long as it contained muriate of lime. Carbonic acid gas was then passed through the solution of submuriate of lime thus obtained, as long as precipitation ensued; after separating the carbonate of lime by filtration, the solution of muriate of lime was decomposed by subcarbonate of ammonia: and the carbonate of lime precipitated, after washing and drying, weighed nearly 94 grains. This experiment was twice repeated without any notable variation in the result. It is therefore evident that the lime necessary to decompose 100 parts of muriate of ammonia, combines with as much carbonic acid as forms with it 94 parts of carbonate; and consequently that rather less than one part of carbonate of lime is required to decompose one of muriate of ammonia, instead of the two parts now employed.

As carbonate of lime is obtained at a low rate, it may be thought unimportant to ascertain how small a proportion of it is capable of effecting the intended purpose: it should however be remembered, that when the requisite quantity is so greatly exceeded as in the present instance, the expense is also increased by the employment of larger vessels and the consumption of more fuel. This salt should have been denominated Ammoniæ Subcarbonas: it is universally allowed to be a subsalt, and its alcaline properties are extremely distinct.

# LIQUOR AMMONIÆ ACETATIS.

As this solution is frequently employed with substances which it is of importance should not be decomposed by

the mixture, the exact point of neutralization should be determined more precisely than by the cessation of effervescence, as directed by the London College. In the Dublin Pharmcopæia it is ordered to be determined by litmus; but as excess of alcali is to be avoided, even more than excess of acid, the saturation should be ascertained by the alternate use of papers stained with turmeric and litmus.

There is one circumstance incident to this solution, which has caused some practitioners to suspect that the acid used in its preparation must have been adulterated by sulphuric acid. It is frequently employed externally with superacetate of lead, and in consequence of the action of these solutions upon each other, considerable precipitation ensues. It may be also suspected that this effect is produced by excess of alcali, but this is not the case, for I have found even when the solution contained acid unsaturated, that the addition of lime water occasioned precipitation of carbonate of lime when more was employed than was sufficient to saturate the acetic acid; and from this experiment it is also evident, that the acetate of lead is decomposed by the carbonic acid evolved from the subcarbonate of ammonia, which remains diffused through the solution.

## LIQUOR AMMONIÆ CARBONATIS.

Dr. Powell has stated in his remarks upon the carbonate of ammonia, that one part of this salt is soluble in two parts of water; now even supposing this statement exact, which however it is not, fl.oz. 16 of water, being equal to about only 15 oz. by weight, would be insufficient to dissolve 8 oz. of the salt, as now directed.

It has been very correctly remarked by Mr. Stocker, [Pharm. Officinal. Brit. p. 122], that water is incapable of

dissolving half its weight of this salt, and I have accordingly found that one pint of water at 60° dissolves rather less than four ounces of subcarbonate of ammonia. From this statement it is evident that this preparation, simple as it is, has been adopted without trial.

On the former method of preparing the Aqua Ammoniæ, Dr. Duncan has very properly observed, [Edinburgh Disp. 2 edit. p. 434], that it is more economical, as well as more scientific, to prepare this solution by dissolving a certain proportion of carbonate of ammonia in water; from which observation the present method has probably been derived.

### LIQUOR AMMONIA.

The Aqua Ammoniæ puræ of the former Pharmacopœia, was prepared by adding two pints of cold, and six of boiling water, to two pounds of lime, and one of muriate of ammonia: from this solution, when cool and clear, one pint was directed to be distilled. This process is not economical, because about one fourth of the solution remains diffused through the excess of lime; and also on account of the small quantity of product obtained in proportion to the quantity of fluid submitted to distillation. The method possessed nevertheless, the indispensible requisite of being practicable, and the merit of simplicity and certainty.

By taking the mean of several experiments, I found the sp. gr. of this preparation to be .954; which is sufficiently concentrated for the usual medicinal or chemical purpose. It must be allowed to be much more important that the mode adopted for obtaining it, should yield a product of certain and equal, rather than of great

strength. When any preparation has been long in use, and its dose consequently generally understood, it seems unadvisable even to improve the method of procuring it, if the alteration necessarily occasions any variation in its strength; in the present instance however, the process has not only been changed for one which is worse, but the medicine has been most materially altered in power.

In preparing the Liquor Ammoniæ, as directed in the new Pharmacopæia, the proportion of lime has been lessened; this is an improvement; but the process as described in the original is impracticable, and when effected by the assistant illustration of the translation, it is on every account inconvenient. The nature of the product too is such, as to expose the community to no inconsiderable danger. This danger is happily diminished by the difficulties and disadvantages which have already been noticed to occur in procuring it.

To obtain this solution, two pounds each of powdered lime and muriate of ammonia, previously mixed, are directed to be introduced into a retort, containing one pint of water; and the ammonia evolved, having passed through a tubulated receiver, is to be condensed by half a pint of water contained in another vessel; a gentle heat is to be applied at first, and afterwards increased until the retort becomes red hot. Of this method the whole is bad. The first objectionable part arises from what seems a principle adopted by the College, viz. that it is more convenient and expeditious to pass a powder than a fluid through a small aperture. When treating of muriatic acid I have noticed this method; but in the present instance it is much more inconvenient, because the mixed powder very soon becoming moist from incipient action, occupies a longer time in passing into the retort; and as-

soon as it reaches the water, the decomposition of the muriate of ammonia being suddenly and greatly increased, much ammonia unavoidably escapes, and the operator is annoyed by being exposed to its pungent action during the introduction of the remaining powder. When a common retort is used instead of a tubulated one, this inconvenience is not only increased, but attended by another. It being impossible to introduce the whole of the powder into the body of the retort, a portion of that which remains in the neck is washed into the receiver as soon as the distillation begins; but having during its stay in the neck been slowly acting, the ammonia absorbs a small quantity of carbonic acid, which decomposing the muriate of lime, renders the product turbid, by the precipitation of carbonate of lime. The evils next occurring are owing to the small quantity of water employed for the solution of the ingredients, and the condensation of the product. In consequence of the tenacity of the solution of a large portion of muriate of lime, it is almost impossible to employ the requisite degree of heat without expelling the contents of the retort into the receiver. In the former preparation the ammonia contained in twelve ounces of the muriate was held by water sufficient to occupy with it a pint measure: in the present method twice as much ammonia is to be condensed by about two thirds of the former quantity of water: by this alteration much ammonia is permitted to escape during the process, and is inevitably lost.

It will be observed that in the original work no directions are given for regulating the evolution, or assisting the absorption of the gas by employing pressure. Having mixed the ingredients in the proportions directed, I applied a gentle heat to the retort, by which the decom-

position of the muriate was suddenly so much increased, and the evolution of the gas rendered so rapid, that to prevent its suffocating effects. I was under the necessity of instantly removing the retort from the sand heat, and of preventing the further decomposition of the muriate of ammonia by plunging it into water. From this it will be readily and rightly concluded, that the retort was destroyed and its contents wasted.

In the remarks upon this preparation, Dr. Powell has directed that the tube of the receiver should dip below the surface of the water contained in the second receiver; and with this precaution I prepared Liquor Ammoniæ possessing the low sp. gr. of .9040.

Among other extraordinary discoveries which have recently been made with respect to ammonia, it might be imagined that its volatility had been observed to have undergone some alteration. In the Pharmacopæia of 1787, the Aqua Ammoniæ puræ was directed to be distilled with "a gentle heat," i. e. from 90° to 100°, whereas in the present, it is judged requisite to heat the retort till it becomes red hot. These directions are the more remarkable because Dr. Powell, in his observations upon this preparation states, that "it parts with its ammonia in the form of gas, if heated to 132°." I do not cite this opinion as correct, for if even this degree of heat were required to volatilize the ammonia, there is no climate on the face of the earth, in which it could be said to possess, under common circumstances, " a strong, peculiar, characteristic odour."

To the inconveniences already ascribed to the new process, may be added Dr. Powell's intimation, that the glass "vessel will of course be broken." This being acknowledged, it is singular that such a method should

have been adopted; because in the last preparation but one, in which distillation is performed, it is stated, that it is "of importance to preserve the vessel;" and since most operators must be of this opinion, I shall presently propose a method by which it may be preserved, as was likewise the case in the process of the late Pharmacopæia.

I have already stated the impracticability of the new process, if followed as described in the original work, and I shall now offer some reasons which will, I think, be sufficient to show, that even with the improvement recommended by Dr. Powell, the method could never have been exposed to previous trial, either by the College, its Committee, or any individual.

From a passage contained in Dr. Powell's preface to his translation, which I have before had occasion to notice, it is to be understood that such processes as really were submitted to examination, were tried at the laboratory of Apothecaries' Hall, and it would also be reasonable to suppose that the products of the new processes had been carefully compared with those of the old.

I have stated that the specific gravity of the Aqua Ammoniæ puræ of the late Pharmacopœia is, .954, but the specimen which I procured at Apothecaries' Hall, under this name, has a sp. gr. of .9906. Now in Dr. Latham's table of doses, that of the Aqua Ammoniæ puræ appears to be twenty drops; which Dr. Powell, as usual converting drops into minims, adopts for the dose of the new preparation. It is however evident, from the great inequality in the specific gravities of these preparations, that their powers must be very different. The extent of this difference I attempted to discover, by referring to Mr. Davy's table of solutions of ammonia, which it may be supposed Dr. Powell has introduced into his translation for the purpose of comparison.

It happens most unfortunately for this conjecture, that .9713, the highest specific gravity, and consequently the weakest solution mentioned in the table, is not so high as that of the old preparation procured from Apothecaries' Hall; and it is equally and additionally unlucky that .9054, the lowest specific gravity, and consequently the strongest solution introduced there, is not so low as that of the new preparation.

According to the table, 100 parts of the solution of ammonia, of sp. gr. .9713, contain about 7 parts of ammonia; whilst 100 parts of the sp. gr. .9054, contain 25; it is therefore evident that the latter solution is more than three and a half times stronger than the former, and it is equally clear, that the Aqua Ammoniæ puræ of Apothecaries' Hall, and the Liquor Ammoniæ of the new Pharmacopœia must differ still more in power. means of an estimate of their respective strengths as correct as I can form by the assistance of the table, the power of the new preparation is to that of the old as 5 to 1. To exhibit therefore equal quantities of ammonia, Dr. Latham's 20 drops should be raised to 100, or Dr. Powell's 20 minims reduced to 4; it is however to be remembered, that in referring to the former preparation, I mean when it has the specific gravity of that which was prepared at Apothecaries' Hall, and which of course must have served the committee as a standard, had any comparison ever been made.

Dr. Powell's translation furnishes another remark, proving almost to demonstration, that the new process was adopted without experiment; he asserts that "the cold produced by the solution of the salt, will counteract the heat produced by the slaking of the lime." I did indeed find that when one pound of muriate of ammonia

was added to half a pint of water, the thermometer sunk from 50° to 30°, but I also found by pouring a similar quantity of water upon an equal weight of lime, that the mercury was raised from 50° to 212°. To render the subject past dispute, I mixed 6 oz. of lime with 6 oz. of muriate of ammonia, and fl. oz. 4 of water, these being one fourth of the quantities ordered for the preparation of the Liquor Ammoniæ: the thermometer immediately rose from 50° to 175°; higher by 45 degrees than the point at which, as I have before observed, Dr. Powell states that "the solution parts with its ammonia in the form of gas."

As the specific gravity of the Aqua Ammoniæ puræ, procured from Apothecaries' Hall, differs so materially from that which I prepared, I shall not insist at all upon the evidence which might otherwise be derived from examining the specific gravity of the Liquor Ammoniæ obtained from the same source. I may however remark, that it is only .9885; differing very little from that of their Aqua Ammoniæ puræ, although its strength ought to be about five times as great.

Upon considering the facts which I have related, and after performing many experiments which it would be tedious and useless to describe, it appeared to me that the inconveniences of the new process might be altogether avoided, by modifying that of the former Pharmacopæia; and that this might be effected in such a manner as to produce a medicine of similar strength: without which indeed, in my opinion, no change could be made with advantage.

In the observations upon Ammoniæ carbonas I have stated that I found 100 parts of muriate of ammonia were decomposable by about 94 of carbonate of lime, and as 100 parts of this substance contain about 56 of lime, 94 will indicate nearly 53 as the quantity of lime required for this purpose. Limestone however, frequently contains other earths; and even when the lime procured by burning it, is sufficiently good to slake upon the addition of water, I have found it to contain about one tenth of carbonic acid: besides it sometimes happens if the lime be not carefully selected, that fragments of carbonate are intermixed with it. On these accounts in the method I am about to describe, I used a greater proportion of lime than is required according to the experiment just alluded to.

Upon 9 oz. of lime I poured half a pint of water, and when it had remained in a well closed vessel for nearly an hour, 12 oz. of muriate of ammonia and about  $3\frac{1}{2}$  pints of boiling water were added to it; the mixture having cooled, the clear solution, obtained by putting the contents of the vessel upon a filter, was divided into two equal portions, and separately distilled without introducing any water into the receiver, and without employing pressure. From one portion I procured fl.oz. 10 of Liquor Ammoniæ, having the specific gravity of .954, which as has been already observed, is precisely that of the Aqua Ammoniæ puræ of the late Pharmacopæia; from the second portion, a similar quantity of product was obtained, scarcely varying in specific gravity from the first.

It is not necessary to enter into a minute description of the advantages which this method possesses over that of the present Pharmacopæia. I shall therefore only add, that every inconvenience which occurs in that process, is obviated in this; and it is to be preferred to that of the former Pharmacopæia, because by employing a small portion of lime so much more of the solution is now saved, that one fourth more product is obtained. It is also more economical in other respects, for by the diminution of the water, smaller vessels and less fuel will answer for the distillation of a given quantity of this preparation.

Liquor Ammoniæ is now directed to be used in many of those preparations in which the subcarbonate was formerly employed; and it is particularly requisite that the attention of practitioners should be directed to this circumstance. I have already noticed the great difference which exists between the strength of the former Aqua Ammoniæ puræ and the present Liquor Ammoniæ; the power of the Linimentum Ammoniæ fortius, and of the Linimentum Camphoræ compositum, has also been greatly increased. It is however much more important to point out the extravagant increase of power added to those medicines which are internally employed, viz.

Spiritus Ammoniæ

..... aromaticus
..... fœtidus
..... succinatus.
Tinctura Valerianæ ammoniata
.... Guaiaci ammoniata

With the exception of the preparation last mentioned, the quantities assigned by Dr. Powell for the doses of these medicines are equal to those of the former weaker preparations given by Dr. Latham. I cannot quit this part of the subject without remarking, that of all the improvements attempted by the College, their failure in this is calculated to produce the most extensive mischief; and I will venture to assert that scarcely one of the preparations above mentioned could be exhibited in the

doses stated by Dr. Powell, without danger to the patient, and disgrace to the practitioner.

#### POTASSÆ ACETAS.

In the former Pharmacopæia this salt was directed to be procured by the repeated addition of small quantities of distilled vinegar to subcarbonate of potash, until the alcali was saturated; and the solution was then to be evaporated to dryness.

In the present Pharmacopæia 1½ pound of the subcarbonate of potash is to be mixed with 8 pints of acetic acid; and to the solution, after evaporation to one half, as much more acid is to be added as may be requisite to saturate the alcali; and the solution, having been again boiled to half, is to be evaporated by the heat of a water bath, until on being removed from the fire it shall crystallize.

I cannot discover why the solution is to be submitted to evaporation before the whole of the acid required to saturate the alcali has been added to it; but even if this be necessary, it does not appear equally requisite to add so small a portion of the acid in the first instance; for the quantity of alcali employed requires about 21 pints of acid to saturate it, of which little more than one third is directed to be used at first.

From the original work it might be imagined that the acetate of potash was to be procured by the usual method of crystallizing salts. In the translation it is however obscurely hinted that it is to be crystallized by fusion; for Dr. Powell, after stating that no directions for crystallizing this salt were given in the former Phar-

macopœia, adds, "It is however a more elegant and uniform preparation, and as it is made by some chemists upon a large scale, it is usually found in the shops; and it requires only the fusion of the salt to give it this appearance;" it is further remarked, "that its crystallization depends upon the liquefaction of the dry salt by heat, and the assumption of a regular form as it cools, rather than on the separation from its solution in water." Wishing however to comply with the obvious directions of the original, rather than to collect information of doubtful accuracy from the translation, I attempted to crystallize this salt by the common mode; and if what Dr. Powell asserts be correct, viz. that water at 60° dissolves an equal weight of this salt, there can be little difficulty attending this operation; but I found that water at 40° dissolved twice its weight, and it was not until the water was reduced by evaporation to about one eighth of its original quantity that I obtained a few crystals, and these could not be detached from the semi-fluid mass in which they were enveloped.

The College have ordered the solution of this salt to be "perfectly neutral;" it is therefore difficult to discover the grounds for Dr. Powell's assertion that in the salt "the alcali rather predominates."

#### POTASSÆ SULPHAS.

It has been shown by deductions from the statements furnished by the table of experiments contained in the translation, upon which I have offered some observations under the head of Acidum Nitricum, that nearly twice the requisite quantity of sulphuric acid has been directed to be employed to decompose the nitre, without any ad-

have conceived that this appearance of waste is removed, by directing the excess of acid to be saturated by the addition of subcarbonate of potash, in order to prepare sulphate of potash.

If the quantity of this salt obtainable from the residuum after the preparation of nitric acid for the purposes of medicine and the arts, were unequal to supply the demand, it would still not be requisite to adopt the very expensive mode of procuring it now directed by the College; but the reverse of this being the case, the employment of too large a quantity of sulphuric acid in preparing the nitric acid is doubly wasteful, for not only the sulphuric acid, but also the subcarbonate of potash required to saturate it, are separately of greater value than the salt prepared by their direct combination.

Supposing the excess of sulphuric acid to be 12 parts, and its cost 48, I find that it would require about 20 parts of subcarbonate of potash, the cost of which would be 240, to saturate it, and these would yield about 21 parts of sulphate of potash, the cost of which, without any allowance for labour and fuel, would be to that of the pure salt made on the large scale, nearly as 2 to 1. From these statements it appears evidently more economical to saturate any unavoidable excess of sulphuric acid by lime, and to reject the sulphate of lime.

Sulphate of potash might have been placed among the articles of the Materia Medica for the same reason that has occasioned the insertion of sulphate of soda.

The statement given by Dr. Powell of the form of this salt when crystallized, is as far as it goes, correct; but through the kindness of the Count de Bournon, I am enabled to present a more detailed and scientific account

than I believe has hitherto appeared, of the crystals of this and some other salts.

The primitive crystal of sulphate of potash is a pyramidal dodecahedron with isosceles-triangular faces; meeting at the summit at an angle of 66°15', and at the base at '113°45'. This form is considerably modified, both by the occurrence of additional faces, and the partial or total disappearance of the primitive ones. The edges formed by the union of the pyramids are very often replaced by narrow planes, forming a short prism: but the crystals more usually become prismatic by the elongation of two opposite faces on each pyramid, rendering the dodecahedron cuneiform; and the crystal then appears to be a long rhomboidal tetrahedral prism of about 66° and 114°, with tetrahedral pyramids, the prism being formed by the four elongated trapezoidal faces, and each of the pyramids by four of the faces of the dodecahedron which remain triangular. Sometimes two of the edges of the prism are replaced each by a plane, and it then becomes hexahedral; and when the same occurrence takes place with the four edges it is rendered octohedral: frequently also both the edges of the pyramid are replaced each by a plane, giving them six instead of four planes. These are not the only forms which this salt assumes, but it would be difficult to give an intelligible description of the rest without figures.

The quantity of sulphate of barytes which 100 grains of this salt yield on the addition of muriate of barytes, is erroneously stated by Dr. Thomson to be 128 grains. I obtained a precipitate which, dried by a red heat, weighed 136.7 grains, being only 1.45 grain more than the proportion mentioned by Kirwan; indicating about 32.8 of sulphuric acid, if with Dr. Thomson, we allow 100

parts of sulphate of barytes to contain 24 of sulphuric acid.

This salt then consits of

32.8 sulphuric acid 67.2 potash and water,

instead of

31 sulphuric acid 69 potash and water,

as stated by Dr. Thomson.

The alteration which has been made in the process for preparing Sulphate of potash, seems to have been borrowed from the Edinburgh Pharmacopæia.

#### POTASSÆ SUPERSULPHAS.

This salt is now introduced into the Pharmacopæia, and it is directed to be prepared by dissolving in water the residuum after the distillation of nitric acid, and setting by the solution, after evaporation, until a pellicle appears on its surface, that crystals may form.

In my observations upon this preparation in the London Medical Review, I have stated that the excess of acid which it contains is in a state of mixture, not of combination; and consequently that this compound cannot be considered as belonging to the class of supersalts. Although this opinion was formed after a careful examination of the salt produced as nearly as possible according to the directions of the Pharmacopæia, yet subsequent opportunities of examination and experiment have shown me that a real supersulphate of potash is obtainable, and consequently that so much of the statement in

the review as tends to establish a contrary opinion, is incorrect.

Some extremely well defined crystals of this salt were prepared and presented to me by Messrs. Howard and Co. of Stratford; their form was distinctly rhomboidal; but as quartz sometimes occurs of this figure, although, as well as sulphate of potash, it is more usually dodecahedral, I conceived it possible that the rhomboid might in this case be derived from the dodecahedron, and consequently that these crystals might be common sulphate of potash. Previously to submitting them to chemical examination, I requested the Count de Bournon to examine their crystalline form, and compare it with that of the sulphate of potash, which he had already described; and this examination showed that the rhomboid differed so materially from that which would be derived from the dodecahedron of sulphate of potash, that these salts must totally differ from each other, either in the nature or proportion of their constituent parts. The primitive crystal of supersulphate of potash, is an acute rhomboid of 74° and 106°, the summits of which are sometimes replaced by planes perpendicular to the axis of the crystal; and when this occurs at the small diagonal of the crystal, it assumes the appearance of an octohedron.

With this information, I proceeded immediately to examine into the causes which had prevented me from obtaining supersulphate of potash on my first attempt. Having dissolved the residual salt in the required quantity of water, after considerable evaporation it was set aside to crystallize, and I readily obtained the octohedral variety of supersulphate just described; a small portion of this being preserved, the remainder was dissolved in the solution from which it had separated, and more water

having been added, and the solution in a slight degree evaporated, I procured only common sulphate of potash: the crystals were indeed externally slightly acid, but the acidity was removed by washing; and the remaining salt, which possessed the usual crystalline form of sulphate of potash, did not redden litmus paper when dissolved in water.

From these experiments it appeared to me that the formation of supersulphate of potash depended upon the degree of concentration of the superfluous acid, and this was ascertained to be the case by the following circumstances. I returned the greater part of the crystals last obtained into the solution which had yielded them, and when they were redissolved, I noted the weight of the solution; and after it had lost about one sixth of its weight by evaporation, I obtained crystals which distinctly possessed the form and properties of supersulphate of potash.

I sent to Apothecaries' Hall for the supersulphate of potash, and upon examining the salt procured, I found that it was at first sour to the taste, and reddened blue vegetable colours; but the acid taste was soon succeeded by a bitter one, and the salt evidently consisted principally of a prismatic variety of the common sulphate of potash, mixed with a considerable quantity of slender prismatic crystals, which, as the Count de Epurnon observed, possessed the form of nitre; from other portions of this salt procured at the same place, I selected many of these crystals, varying from a quarter of an inch to an inch in length. Being unwilling to trust merely to external characters in proving a circumstance of some importance, I added a portion of this salt to sulphuric acid in a retort, and by the application of heat I obtained a very considerable quantity of nitric acid. The examination of this salt merely proved, that the supersulphate of potash was not produced because the quantity of sulphuric acid used was insufficient even to decompose the nitre, and that the undecomposed portion consequently crystallized with the sulphate of potash.

It is indeed true that even this mixed salt contained, as already mentioned, a slight excess of sulphuric acid in mixture with it; and I shall now state the proportion of dried subcarbonate of soda required to saturate the acid of equal quantities of the true supersulphate of potash; of the salt with excess of acid, in a state of mixture; and of that procured from Apothecaries' Hall.

One hundred grains of supersulphate saturated 25 grains of dried subcarbonate of soda; 100 grains of sulphate mixed with excess of acid saturated about half a grain; and a similar quantity of the salt from Apothecaries' Hall, saturated about one grain.

The result of the examination of the last mentioned salt, is not the only circumstance which induces me to suppose that neither the College nor their Committee have examined the true supersulphate of potash. Dr. Powell observes that "on crystallizing, it chiefly fixes itself to the side of the vessel, from which bed slender needles sometimes shoot." I have never yet seen supersulphate of potash in needle-form crystals, although it may undoubtedly have occurred; and in the only variety of the rhomboid which I have observed, the crystal is rendered shorter instead of becoming prismatic. Dr. Powell had probably seen the article prepared at Apothecaries' Hall, his description of the supersulphate being such as he might be expected to give of that mixed salt.

The next circumstance which I shall state in corroboration of this opinion is, that Dr. Powell gives two

drachms as the dose of supersulphate of potash. We are informed in his remarks upon Acidum Sulphuricum dilutum, with a degree of accuracy almost unparalleled in the translation, that a fluidounce of this preparation saturates about 107 grains of dried subcarbonate of soda, and its dose is stated to be 40 minims; consequently the utmost dose of diluted sulphuric acid saturates about 9 grains of dried subcarbonate of soda, whilst it follows from what I have stated of the saturating power of supersulphate of potash, that two drachms of it neutralize 30 grains of dried subcarbonate; so that the dose of sulphuric acid in the supersulphate of potash, is more than three times as great as that in the diluted sulphuric acid. It ought also to be remembered, that the quantity of sulphuric acid given by Dr. Powell, is nearly one half greater than that directed by Dr. Latham, and consequently that the dose of sulphuric acid which two drachms of supersulphate contain, is about five times as great as that contained in 40 drops of the dilute sulphuric acid of the former Pharmacopæia, which quantity is stated by Dr. Latham as its utmost dose. delinerate our our bearings

From the uncertainty which attends the attempts to procure real supersulphate of potash, it appears to me extremely probable, that some practitioners who may have obtained slightly acid sulphate instead of the real supersulphate, may have exhibited it in much larger quantities than it would be proper to give of the true salt, and consequently if the supersulphate were afterwards procured, it may have been used in very improper doses. From the large quantity of nitre which the mixed salt I have described contains, very unexpected effects may have been produced; and its true nature being unknown, these effects may have been attributed to causes

which had no share in their production. These considerations rendered it important to ascertain the proportion of nitre mixed with the supersulphate of potash obtained at Apothecaries' Hall. For this purpose I dissolved two portions of 25 grains each of that salt in distilled water, and added muriate of barytes to the solutions as long as precipitation ensued; the sulphate of barytes thus obtained, after washing and drying, weighed in one experiment 19.5, and in the other, 20.5 grains, giving a mean of 20 grains; consequently 100 grains of the salt would give 80 of sulphate of barytes: I have mentioned in page 38, that 136.7 of this substance indicate 100 of sulphate of potash, 80 will accordingly give about 58. One hundred grains of this mixed salt consist of nearly

doing bus a sol42 nitrate of potash. In a doing visitate

When, therefore, 2 drachms of this compound are exhibited, instead of the like quantity of supersulphate of potash,  $2\frac{1}{2}$  scruples of nitre are given; exceeding by one scruple, the largest dose mentioned by Dr. Powell.

In order to diminish the uncertainty attendant upon the production of supersulphate of potash, I may state, that when the residual salt is dissolved in only about an equal weight of water, the solution on cooling deposits supersulphate of potash, without the occurrence of the slightest pellicle, which is directed in the Pharmacopæia to be observed before the solution is removed from the fire. I find moreover, that when the solution is evaporated only until this appearance takes place in a very trifling degree, it becomes on cooling a perfectly solid crystalline mass. I am not however prepared to assert, that supersulphate may not be obtained when the solution is weaker than

I have mentioned, or that if it be evaporated until a pellicle begin only to appear, it will invariably become solid on cooling. I have only to observe, that in following the directions of the College, I did not succeed, whilst, omitting them, my success was complete.

The reason of the introduction of this salt into the Pharmacopæia is stated by Dr. Powell to be, that "it affords a useful means of exhibiting sulphuric acid in a solid form." I apprehend however that considerable difficulty will occur in ascertaining an eligible method of giving it. The extreme acidity precludes its being exhibited in the form of an electuary or in solution; the latter method must also be considered inadmissible, as rendering nugatory the only reason assigned for the employment of this compound; and I find that two drachms of this salt cannot be made into fewer than about forty pills; a quantity which is much too large for one dose; and which there are not perhaps two practitioners who would prescribe, or one patient who would take.

# POTASSE TARTRAS.

petash; alescraples of nitre are given; exceeding by one

scruple, the largest dose mentioned by Dr. Powell

Among other remarks upon Potassæ Acetas, I have noticed the impropriety of not determining the quantity of alcali requisite for the saturation of an acid by precise means; the same observation is equally applicable to the method ordered to be employed for obtaining the salt now under consideration.

I find that 36 parts of supertartrate of potash require 15.7 parts of subcarbonate of potash for their saturation, instead of 12 parts as directed by the College; it is however evident that the proportion of the alcali required for this purpose, must vary with the different degrees of dryness of the subcarbonate of potash employed.

The form of the primitive crystal of supertartrate of potash, is a rectangular octohedron, having two of its faces more inclined than the other two; the former meeting at the summit at an angle of about 60°, and at the base at about 120°; the latter at the summit at 50°, and at the base at 130°. When the superabundant acid has been saturated so as to produce Potassæ Tartras, the primitive form is altogether different; it is a rectangular tetrahedral prism, of which the height being one, the sides of the terminal faces will be to each other as 4 to 5; most commonly the summit is dihedral from the replacement of one of the solid angles of the primitive crystal by a plane which makes with the terminal face an angle of 162°38': frequently the triangular faces of the pyramid become very narrow trapezoidal faces, by the increase of two opposite planes; and this is one of the more usual varieties of this salt.

The process for preparing this salt remains nearly the same as in the late Pharmacopæia.

#### POTASSÆ SUBCARBONAS.

The only change to which the method of preparing this salt has been subjected in the new Pharmacopæia is, that 3 pounds of the impure salt are to be dissolved in 3½ pints of water, instead of 4½ pints as previously directed; this diminution of the quantity of water is certainly an improvement, for which the College are perhaps indebted to the Edinburgh or Dublin Pharmacopæia.

The substance from which subcarbonate of potash is

of nomenclature in its progress through the Pharmaco-pœia. In the Materia Medica it is called Potassa impura, which is explained to be Carbonas Potassæ impura; and yet without either the addition or abstraction of carbonic acid, it becomes by solution in water and evaporation to dryness, Potassæ Subcarbonas: the only inaccuracy therefore which the College retained the power of committing, was to have denominated it Supercarbonas Potassæ impura.

In the translation, Potassa impura is literally rendered impure potash: the potash and pearl-ash of commerce differ however very materially, the former is much more impure and caustic than the latter, and yields by solution and evaporation a salt of a much darker colour; and although it would have been improper to have translated Potassa impura by pearl-ash, yet this word ought also to have been employed as explanatory of the intention of the College.

When pearl-ash is used for preparing subcarbonate of potash in the mode now directed, I find that 100 parts of the product consist very nearly of

26 carbonic acid

71 potash and water

2 muriate of potash

I sulphate of potash, and a little silica.

This preparation possesses undoubtedly a sufficient degree of purity for any medicinal purpose: it must therefore be considered superfluous and expensive to direct it to be obtained by the decomposition of tartar; and if there be any instances in which it is less requisite to employ a pure alcali than in others, those very instances have been selected by the College.

#### POTASSÆ CARBONAS.

This preparation is now introduced into the Pharma-copœia: and the College, in order to saturate subcarbonate of potash, have adopted Berthollet's very expensive method of procuring carbonic acid, by decomposing subcarbonate of ammonia. It is much more economical to saturate the subsalt with carbonic acid, evolved during the decomposition of carbonate of lime by sulphuric acid; and the operator might at least have been suffered to employ this process without the fear of legal chastisement.

The expense of the present method is also unnecessarily augmented by directing subcarbonate of potash from tartar to be employed, instead of the salt obtained by the common method; for even if its impurities much exceeded those which actually exist, crystals of carbonate of potash are easily distinguishable from those of sulphate or muriate of potash.

## LIQUOR POTASSÆ SUBCARBONATIS.

In the late Pharmacopæia this solution was directed to be obtained by the tedious method of exposing subcarbonate of potash to the atmosphere until it deliquesced. It is now to be prepared by the preferable mode of direct solution of the salt in water. This improvement was perhaps suggested by the Lixivium mite of the Dublin Pharmacopæia of 1794.

## LIQUOR POTASSM.

To prepare this solution, one pound of subcarbonate of potash dissolved in six pints of boiling water, is to be added to an equal weight of lime, upon which two pints of water have been previously poured.

I have mentioned that 100 parts of subcarbonate of potash contain nearly 26 of carbonic acid, requiring 33 parts of lime for their saturation; so that 12 oz. of subcarbonate require only about 4 oz. of lime for the purpose of abstracting the carbonic acid, instead of an equal quantity as now directed. For the reasons advanced when treating of Liquor Ammoniæ, a larger quantity of ordinary lime than is strictly requisite, may be advantageously employed, but the great excess of this substance directed by the College, when mixed with the carbonate of lime necessarily formed during the process, occasions so considerable a residuary mass, that according to Dr. Powell, nearly one third of the solution remains diffused through it.

I find that half the proportion of lime now directed, is capable of rendering potash sufficiently caustic for medicinal purposes; whilst so great is the difficulty of abstracting completely the carbonic acid from subcarbonate of potash, that it is not effected by twice the quantity of lime used in the present process.

When Liquor Potassæ, containing a small quantity of carbonic acid, is mixed with one eighth of its bulk of lime water, no immediate, and scarcely any ultimate effect is produced; but when a larger portion of limewater is used, precipitation of carbonate of lime immediately occurs. The first mentioned circumstance shows, either that carbonate of lime is not formed, or that

it is decomposed by the great excess of potash; and I have some reason for believing that the latter is the fact.

This process has been improved by omitting the boiling, as in the Dublin and Edinburgh Pharmaco-poeias.

#### SODA TARTARIZATA.

No material alteration has been made in the process for procuring this salt: it is directed to be prepared by adding 24 parts of supertartrate of potash, to 20 parts of subcarbonate of soda; whereas I find that only about 18 parts of the alcaline subsalt are required for this purpose. The quantity must however be subject to variation, on account of the efflorescent property of subcarbonate of soda.

Dr. Powell has described the crystals of this salt to be prisms of eight or ten unequal sides; this description is not incorrect, but incomplete. The primitive crystal is a right rhombic prism of 80° and 100°; the edges of the prism are differently replaced so as to render it sometimes hexahedral, or octohedral; but more frequently decahedral, or dodecahedral.

In the Pharmacopæia of 1787, the name of Soda Tartarizata was admissible; but to continue the term tartarizata is improper, because it is deriving the specific name of a compound from one which the ingredient formerly conferring it no longer retains. Dr. Bostock, in his remarks upon the nomenclature of the New London Pharmacopæia has truly stated, that "the term Soda Tartarizata is confessedly unscientific, and it is adopted by the College, because, as Dr. Powell observes, it has been thought more convenient than the proper appellation, Tartras

Sodæ et Potassæ; the word tartarizata is thus by the most arbitrary and unauthorized assumption made to express the fact, that this salt contains potash!"

#### SODÆ SULPHAS.

The superfluous acid of the salt remaining after the distillation of muriatic acid, was directed in the late Pharmacopæia to be expelled by heat. This method was certainly ineligible, but that substituted for it, which is probably borrowed from the Dublin Pharmacopæia, is very uneconomical; although less so than the process for preparing sulphate of potash, which it resembles, because the superfluous acid to be saturated by the addition of subcarbonate of soda is not in such great excess. The more economical method which I have recommended when treating of sulphate of potash, is equally applicable in preparing the salt now under consideration.

The primitive crystal of this salt appears to be a right rhombic prism, of about 72° and 108°. It is frequently difficult to distinguish the planes of the prism, on account of their being deeply channeled. The terminal faces of these crystals are often replaced by a dihedral summit with triangular faces, occurring on the edges of 72°; and this variety has probably given rise to the opinion, that the primitive form of this salt is a cuneiform octohedron; but as the planes of the summit make with the edges of 72° an angle of about 137°, they meet each other at 86°; whereas in the supposed crystal, it would be 108°. The pyramid also frequently becomes hexahedral, by the occurrence of four additional faces upon the edges of the terminal faces, with which they form an angle of about 125°.

#### SODÆ SUBCARBONAS.

In the former Pharmacopæia two pounds of barilla were directed to be boiled in one gallon of water; in the present, the quantity of water has been very unnecessarily doubled. This alteration is the more extraordinary, because it may be inferred from the remarks which Dr. Powell has made upon this preparation, that about one tenth part of the quantity of water directed to be employed for the solution of the soluble part of one pound of barilla, is more than sufficient to dissolve one pound of the pure salt.

The same note also informs us, that two parts of cold water will dissolve one of subcarbonate of soda: of course, if this be true, it will be useless to attempt to obtain it by the method directed by the College; for the soluble part of one pound of the impure salt is directed to be set to crystallize, when dissolved in more than two pounds and a half of water.

The primitive crystal of this salt, as described by Romé de Lisle, is an octohedron with a rhombic base of 60° and 120°; the planes of which meet at the summit at 104°, and at the base at 76°: this crystal varies by becoming cuneiform; and also by the replacement of the solid angle of the summits by planes parallel to the base; this decahedral variety is the most common.

#### SODÆ CARBONAS.

This salt is now introduced into the Pharmacopæia. The quantity of subcarbonate of ammonia directed to be employed in the preparation of it is unnecessarily large;

and the process, apparently suggested by that for producing Potassæ carbonas, has been adopted without proper consideration. In 100 parts of the crystals of subcarbonate of soda, according to the authorities quoted by the translator, about 21 parts of soda are combined with about 14 of carbonic acid; and in the carbonate of soda, the acid and alcali are in equal quantities: therefore to convert subcarbonate into carbonate, 7 parts of carbonic acid must be added. Now I find that 100 parts of subcarbonate of ammonia yield about 50 of carbonic acid; therefore 100 of subcarbonate of soda require for saturation the acid of about 14 of subcarbonate of ammonia, instead of 25, as directed in the Pharmacopæia.

#### ALUMEN EXSICCATUM.

When alum is exposed to a considerable degree of heat, it loses not only the greater part of its water of crystallization, but frequently a portion of the salt is decomposed, and rendered insoluble, although some of it remains in the state of supersulphate.

The exsiccation of alum therefore unless carefully conducted, must be considered not merely as useless, but as rendering a substance which in its usual state is of an unvarying degree of strength, very liable to be diminished in power instead of being increased, as appears to be intended by its exsiccation.

The name of Alum is absolutely unchemical; and if it be retained on account of its convenience, and not being liable to be misunderstood, the same reasons might have induced the College to have retained the appellations of Nitre and Tartar.

#### CALX.

It is always easy to procure good lime; and as it would be impossible to detect any difference in the effect produced by lime obtained in the usual way, and that prepared by chemists, when well burnt; the directions for making it now introduced, must be reckoned not only superfluous but oppressive, many chemists being so circircumstanced that it is not in their power to comply with the mandates of the College.

### CRETA PRÆPARATA.

In conformity with the chemical nomenclature adopted by the College, this substance ought to have been denominated carbonate of lime. It is now directed to be prepared by elutriation instead of levigation. As coral and crabs' claws are rejected from the Pharmacopæia, it is singular that oyster shells should have been retained. Dr. Powell indeed states, that shell is a purer carbonate of lime than chalk; but as it appears by his table of doses, that they may be exhibited in equal quantities, chalk ought to be preferred on all occasions; because, on account of its friability, it is more easily reduced to a fine powder.

#### MAGNESIÆ CARBONAS.

This preparation is probably a mixture of carbonate and subcarbonate of magnesia. The quantity of subcarbonate of potash employed to decompose the sulphate of magnesia is considerably too large, for I find that three parts only of the alkali are required instead of four, for the decomposition of four parts of the sulphate.

### SULPHUR PRÆCIPITATUM.

In the late Pharmacopæia this substance was directed to be obtained by adding sulphuric acid to sulphuret of potash, composed of one part of sulphur and five of subcarbonate of potash: instead of this, solution of sulphuret of lime prepared as in the Pharmacopæia of 1745, is now directed to be decomposed by muriatic acid. This change has been made, because according to Dr. Powell, the precipitate procured by the method directed in 1787, contained sulphate of potash. That this may happen when the product is not sufficiently washed is probable, but it is absolutely incredible that it should ever have been so clumsily prepared, as that the precipitate "owed a good deal of its whiter appearance" to this circumstance.

It appears to be merely a question of economy whether the new method is preferable to the former; and consequently to be decided by comparing the cost of the solvent and precipitant of the sulphur employed in each process. In making this comparison, I shall assume that the products and expense of labour and fuel are equal in both cases; although it must be admitted that some sulphur is lost during fusion in the former method, whilst no waste is attributable to the boiling employed in the present.

Process of 1787.	,	
10 parts of sulphur, estimated	at .	. 5
50 subcarbonate of potash		. 60
30 sulphuric acid		. 12
total cost	t-	77

## Process of 1809.

to	parts of sulphur, estimated at	5
30	lime	I
17	muriatic acid	17
	total cost	23

The first statement requires no explanation, the proportion of sulphuric acid necessary to saturate the alcali of a given quantity of subcarbonate of potash having been already mentioned at page 37. To ascertain the proportion of lime which sulphur is capable of dissolving, I boiled 50 parts of the former with 150 of the latter; 38 parts of sulphur remained undissolved, and this experiment was repeated with scarcely any variation in the result. Then as 112 parts of sulphur take up 50 of lime, 10 parts will dissolve nearly 41 parts. At page 10, I have stated that 16.43 parts of carbonate of lime require 35 of muriatic acid of sp. gr. 1.174, to decompose them; and 100 parts of carbonate containing nearly 56 of lime, 16.43 will give 9.2 as the quantity of lime saturating 35 of muriatic acid; consequently 4.5 will require about 17 parts for the same purpose.

It appears from this statement that the new process is more economical than the former in the proportion of more than 3 to 1; it is however capable of further improvement by diminishing the proportion of lime, the quantity directed being about five times more than is necessary.

I have minutely investigated the changes effected in this process, because I believe that it presents the only opportunity of announcing a decided improvement unborrowed from the obvious and ordinary sources: and it is but justice to observe, that so far as my researches have extended, the substitution of muriatic for sulphuric acid appears to have originated with the College.

## ANTIMONII OXYDUM,

ET

#### ANTIMONIUM TARTARIZATUM.

"All the antimonial preparations," observes Dr. Powell, "have undergone a considerable revision, and the present oxyd by precipitation, is substituted for three which the former Pharmacopæia contained, Antimonium calcinatum, Antimonium vitrifactum, Crocus Antimonii;" and, apparently more fully to impress the mind of the reader with the magnitude of the improvement, the following names are affixed as synonyms:

It is moreover evident that Dr. Powell considers this oxide as similar to the pulvis Algarothi, called in the Pharmacopæia of 1720, mercurius vitæ; for when

sulphuret of antimony is treated as now directed, he states that "the first product is muriate of antimony," which, "if poured into water precipitates its oxyd, and this has been kept under the name of pulvis Algarothi."

The following are the directions for preparing this oxide:

"Take of Sulphuret of antimony powdered, two ounces;
Muriatic acid, eleven fluidounces;
Nitric acid, one fluidounce.

The acids being mixed together in a glass vessel, add the antimony gradually thereto, and digest em in a boiling heat for an hour; then strain the solution, and pour it into a gallon of water in which two ounces of subcarbonate of potash have been previously dissolved; wash the precipitated powder by repeated affusions of water, until all the acid is washed away; then dry it upon bibulous paper."

Diminution of the number of processes is perhaps one of the most desirable alterations which a Pharmacopæia can undergo: if then this oxide of antimony may be substituted for all those which Dr. Powell says that it resembles, a better reason for adopting it could not be assigned. But after alluding to some improvement, which however is not very discernible, he informs us, "that another advantage to be expected from the present process, is its uniform ty, compared to those where high temperatures are employed, in which the product varies, from a number of circumstances influencing the regular continuance of its degree." The validity of these statements I shall hereafter examine.

In order to ascertain the nature of the changes now made in the preparations of antimony, it will be necesThis is described in most of the treatises on chemistry, but I shall refer principally to Dr. Duncan's dispensatory, and Dr. Aikin's edition of Dr. Lewis's dispensatory. There are other reasons besides Dr. Powell's suggestions for the improvement of the first mentioned work, for supposing that he is not unacquainted with it; and the latter he particularly recommends: nevertheless, there are in these works observations of the highest importance respecting antimony, which he seems to have entirely overlooked.

Dr. Duncan states that the preparations of peroxide of antimony "are found to be perfectly inert when taken into the stomach," whilst those in which it is in the state of protoxide "act in very minute doses;" [2d. edit. p. 162.] and it is distinctly expressed that the antimony contained in the glass and crocus, and also in the pulvis Algarothi, is in the state of protoxide; and further, that the diaphoretic or calcined antimony is a peroxide.

In confirmation of the opinion that diminished power is consequent upon increased oxidizement, I shall refer to Dr. Latham's table of doses. In this it appears that the largest dose of glass of antimony is two grains, and of the crocus only one; whereas of the calcined antimony, sixty grains may be exhibited.

The bezoarticum minerale was prepared in the Pharmacopæia of 1720, by treating the mercurius vitæ with nitric acid so as to convert it into peroxide. With respect to these preparations, Dr. Aikin states [Lewis's dispensatory, p. 107] that the mercurius vitæ "operates in the dose of two or three grains, as a most violent and dangerous emetic:" but when it is converted into bezoarticum minerale, it becomes "equally inactive" with the oxides of

antimony "prepared by deflagrating crude antimony with twice or thrice its weight of nitre."

As this oxide of antimony is described by Dr. Powell as being similar to substances which are unlike each other, we are at a loss to ascertain which it is meant to resemble. This difficulty is apparently obviated by the assertion that "the precipitate is in the form of a white powder, and it constitutes the minor oxyd of Proust, which is composed of antimony 81.5, oxygen 18.5:" and it appears by Dr. Powell's table of doses, that ten grains of this protoxide may be given without any emetic effect.

According then to Dr. Powell's statement, protoxide and peroxide of antimony are synonymous, and ten grains may be properly exhibited; but if we combine these assertions with those of the authorities which he recommends, it appears that, whilst ten grains of the precipitated oxide are a safe dose, two are "a most violent and dangerous emetic," and sixty grains "perfectly inert;" and consequently, that two are much more than ten; and sixty much less. Well indeed has Macquer observed, that some physicians have considered this preparation as occasioning "accidents so terrible," that instead of being called the mercury of life, it ought to have been denominated the mercury of death!

After all that has been said of the extreme impropriety of employing this oxide, I am disposed to think that the method of preparation is, if possible, worse than the medicine itself. It has not a single property essential to a good process; whilst every property which it does possess is more than sufficient to stamp it bad. It is peculiarly unfortunate, that of uniformity, the very property for which it is said to have been introduced, it is abso-

lutely destitute; and I will venture to assert without fear of contradiction, that if Dr. Powell have tried the process, some of the properties which he attributes to the product, must have arisen from his procuring what it was not his intention to obtain, otherwise he would not have ventured to recommend ten grains of this oxide as a dose, nor would have asserted, that when two parts of it are boiled in a solution of three parts of tartar, "more oxyd than may be strictly necessary is directed."

I shall now proceed to state the various objections to which this process is liable. The least of them is the annoyance to which the operator is constantly exposed, by the evolution of much noxious vapour during the decomposition of the sulphuret of antimony. In some chemical processes attended with extrication of vapour, such for instance as dissolving silver or mercury in nitric acid, the operator may add such a portion of the metal to the acid as will permit him to leave it for a considerable length of time without making addition. In the present process however, the circumstances are widely different; the action of the acid upon the sulphuret is so rapid as to require continual attendance: for if the antimony be not very gradually added to the acid, violent action ensues, accompanied with the sudden evolution of vapour, and frequently with the dispersion and total loss of the ingredients.

In the next place, it is not adapted for the preparation of such large quantities of oxide as are required for forming tartarized antimony; one person cannot conveniently manage several processes on the small scale at once; and as the sulphuret of antimony can only be added to the acid in small portions at a time, no advantage would result from the employment of a large quantity of acid in one operation.

The next objection which I shall make to this process is, that extravagance is added to costliness. When two ounces of sulphuret of antimony are treated with eleven fluidounces of muriatic, and the requisite quantity of nitric acid, one ounce and three quarters of submuriate of protoxide of antimony are obtained by decomposing the solution with water: but when four ounces of the sulphuret are boiled in eleven fluidounces of muriatic acid, and a sufficient proportion of nitric, two ounces and three quarters of submuriate are obtained by precipitation. These experiments show that the muriatic acid directed for the decomposition of two ounces of sulphuret of antimony is capable of decomposing more than three ounces of this compound. Of the nitric acid ordered one eighth is sufficient, and I shall shortly have occasion to notice the important effects produced by the excess. The boiling, to which the acid is directed to be submitted after the antimony has been dissolved in it, is productive of no advantage; and I am apprehensive that some part of the uncertainty attendant upon the process may be derived from this circumstance. This I shall consider more at length hereafter.

I have already advanced some reasons for supposing that Dr. Powell has never obtained the protoxide of antimony by employing the process of the new Pharmacopæia, and I shall now state one which seems even to prove that he never tried the process at all. When mentioning the pulvis Algarothi, he says, "Bergman considers this precipitate to be a pure oxyd; but it is doubtful whether it be entirely free from muriatic acid, therefore the separation of this is secured if any be present, by adding the subcarbonate of potash, though some nevertheless still consider it as a submuriate." So far however is this

security from being attained, that I found one-fourth part of the solution more than sufficient to neutralize the subcarbonate of potash, intended for the saturation of the whole of it: the College indeed seem to have had a presentiment that the means directed for the separation of the muriatic acid from the oxide of antimony would prove incapable of effecting it; otherwise they would have undoubtedly employed the word "salt" instead of "acid," in directing the washing of the precipitate. As however submuriate of antimony answers perfectly well for the preparation of tartarized antimony, the insufficiency of the means prescribed by the College, for the separation of the muriatic acid, serves only to prove the advantage which experiment possesses over conjecture.

In the London Medical Review, I have stated my inability to procure by this process an oxide of antimony, capable of forming tartarized antimony by the usual method of boiling in a solution of supertartrate of potash: and these experiments, together with Dr. Powell's statement that ten grains of this oxide were not emetic, induced me to assert as my opinion, that from such an oxide it was impossible to procure emetic tartar. It is true in fact, as it is consistent with theory, that an oxide of antimony which does not possess emetic power, cannot confer it.

I have since found that I am not the only person to whom this difficulty has occurred, Mr. Stocker, of Guy's Hospital, having informed me, and indeed he has since laid the results of his experiments before the public [vide Pharm. offic. Brit. article Antim. tart.], that he had been foiled in every attempt to procure tartarized antimony by employing the antimonii oxydum, now directed:

and it appeared that the difficulties which he had experienced were precisely similar to those that had occurred to me: these difficulties I shall now again relate.

Upon treating the sulphuret of antimony as directed, I obtained a precipitate, which, after having been dried in a moderate degree of heat, was boiled in a solution of the quantity of tartar ordered by the College. A very small portion of the oxide appeared to be dissolved, and the insoluble part passed the filter, rendering the solution extremely turbid: indeed I found it impossible to obtain a clear solution, either by giving the oxide an opportunity of subsiding, or by repeated filtration through many folds of paper. This experiment was made several times without any variation.

At length however, Mr. Stocker informed me that Mr. South, jun. had succeeded in obtaining tartarized antimony by following the directions of the London College; and the mere inspection of the salt which he had procured sufficiently proved this to have been the case. On stating the difficulty and defeat which I had repeatedly experienced, Mr. South kindly assented to my proposal of conducting the process together, and it was agreed that each should employ the method he had previously adopted. Mr. South accordingly made the experiment in a vessel having a long neck, whilst I employed a shallow basin. Upon examining the oxides procured by decomposing the solutions with water, we found that the precipitate obtained by the first method was perfectly and readily dissolved by a boiling solution of tartar, and produced by evaporation crystals of tartarized antimony; whereas that procured by the latter process was nearly insoluble when similarly treated, the solution passing the filter turbid with undissolved oxide.

It was evident from Mr. South's statement that he had almost as constantly succeeded when employing a narrow mouthed vessel, as I had failed when using a shallow one: and I have found the difference thus occasioned to be extremely great; for when the solution is boiled in a flask, about one twenty-fourth part only of the acid is evaporated, whereas when a basin is employed, only one-sixth of it remains.

These facts induced me to suspect that by boiling the acid for an hour after the antimony is dissolved in it, as directed, the superfluous nitric acid is so concentrated by the evaporation of the water, when a shallow vessel is employed, as to be decomposed by the protoxide of antimony; this consequently becoming peroxide. My suspicion was strengthened by finding that when I followed the process of the Dublin Pharmacopæia, in which only a fluidrachm of nitric acid is employed instead of a fluidounce, I never failed to procure protoxide of antimony, although I evaporated the solution even more than in the experiments which I have made upon the process of the London College.

After quoting Dr. Duncan's remarks, it may seem unnecessary to adduce further evidence to prove that peroxide of antimony cannot be used for preparing tartarized antimony: the fact is however so clearly stated by Thenard [Ann. de Chim. tom. 41. p. 53], that I shall likewise introduce his opinion. Speaking of antimony he observes, "s'il est très-oxidé, comme l'est l'oxide d'antimoine au summum d'oxidation qui se trouve dans l'antimoine diaphoretique, il ne se forme pas d'emetique." In confirmation of this statement I may add, that I found, upon treating antimony with nitric acid, that 100 parts of it combined with 31.25 of oxigen; and by

boiling 100 parts of this oxide in a solution of 150 parts of tartar, only 27.5 were dissolved, and the crystals obtained by evaporation did not contain any oxide of antimony.

Mr. Brande having acquainted me that he had experienced difficulties exactly resembling those which I have described, and apparently resulting from the same cause, I mentioned to him the circumstance of the difference produced, seemingly, by the form of the vessel employed, and the success which I had witnessed when following the method adopted by Mr. South. I also informed him that on repeating these experiments I had experienced results almost diametrically opposite to those first produced: i.e. when conducting the process in a shallow basin I procured protoxide of antimony, whereas with the flask I obtained a mixture of both oxides.

When I again repeated the experiments with the assistance of Mr. Brande, using a flask, we obtained protoxide of antimony mixed with a little peroxide; and afterwards, in two similarly conducted experiments, I obtained protoxide. We also ascertained by numerous experiments, that any oxide of antimony with which tartarized antimony could be prepared, uniformly received an increase of weight by treatment with nitric acid; whereas that which could not be so employed, suffered no change by it.

Having thus demonstrated that the action of the nitric acid was the cause of the difficulties which had occurred, it remained only to decide whether in the greater number of instances, the failure was attributable to the increased evaporation, and consequent concentration, resulting from the employment of a shallow vessel. With this view I performed ten additional experiments, the first five of which were made in open vessels of broad surface, in which from twelve fluidounces of the

mixed acids I obtained, upon an average, two fluidounces of muriate of antimony; whilst in the five following experiments, using long necked vessels, I procured generally about eleven fluidounces and a half of that solution.

To examine the products of these experiments, and to determine whether they contained any peroxide of antimony or not, I boiled the several precipitates in solutions of tartar; as from the known insolubility of peroxide of antimony in certain states of aggregation, any portion of them which is insoluble in more tartar than is requisite to dissolve protoxide of antimony, must be peroxide: for the protoxide contained in glass of antimony being readily dissolved in solution of tartar, it may be safely concluded that the less firm state of aggregation in which it occurs in the precipitate, cannot prevent its being dissolved by similar means.

## In a Shallow Vessel.

Exper. I. Colour of the precipitate, yellow: two parts of it were not nearly dissolved by being boiled in a solution of three parts of tartar: the fluid passed the filter mixed with the insoluble oxide, and remained turbid.

Exper. II. Colour white. One hundred parts being boiled in a solution of fifty parts of tartar, the filtered solution gave crystals of tartarized antimony: nearly 40 parts of the oxide were dissolved, and the remaining 60 having been boiled in a solution of twice their weight of tartar, 15 parts remained undissolved. It appears therefore by this experiment, that 100 parts of the precipitate contained at least 15 of peroxide, and probably much more: for peroxide of antimony is not, as has been asserted by Dr. Duncan, insoluble in acids; its solubility depending, like that of peroxide of iron, upon its state of aggregation. When the oxide described in this statement was

moist, tartar dissolved more than half its weight of it; but the precipitate procured by decomposing this solution with ammonia, when dried, was not dissolved by being boiled in a solution of four times its weight of tartar.

Exper. III. Colour, yellowish white. One part of this submuriate was not dissolved by ebullition with a solution of four parts of tartar. The solution passed the filter clear. Three parts of it being boiled in a solution of two parts of tartar, some crystals of tartarized antimony were procured by evaporation. This product therefore, as well as the preceding one was a mixture of protoxide and peroxide.

Exper. IV. Colour, yellowish white. This precipitate was evidently more insoluble in a solution of tartar, than the last. A boiling solution of four parts appeared to dissolve but a very small portion of two parts of the submuriate; and when two parts of it were boiled in a solution of one part of tartar, the solution passed through the filter turbid with undissolved oxide: on this account I could not determine that it contained any protoxide.

Exper. V. Colour yellow. One part of this precipitate was not dissolved by ebullition in a solution of four parts of tartar; but when two parts were boiled in a solution of one part of tartar, a small quantity of tartarized antimony was obtained.

# In a Flask.

Exper. VI. Colour white. Insoluble by ebullition in a solution of four times its weight of tartar: the solution passed the filter extremely turbid with undissolved submuriate. I could not therefore ascertain that any part of the precipitate was in the state of protoxide.

Exper. VII. This product exactly resembled the preceding one. Exper. VIII. Two parts of this precipitate were not dissolved by boiling in a solution of three parts of tartar. I did not examine whether it contained any protoxide.

Exper. IX. Yellowish white. A solution of three parts of tartar dissolved, by boiling, a very small portion of two parts of this precipitate.

Exper. X. Colour yellowish white. Insoluble by boiling in a solution of four times its weight of tartar.

Since then these precipitates were all insoluble in larger proportions of tartar than are required for the solution of protoxide of antimony, it is evident that I did not in any one instance succeed in obtaining protoxide of antimony, unmixed with peroxide; and in several instances I have reason to believe I obtained the latter oxide only.

It will not be necessary to recapitulate the variations produced in these experiments when similarly conducted, or to point out the cases of resemblance resulting from the employment of dissimilar means; it is evident that the conjecture I had formed as to the cause of the production of peroxide was entirely erroneous, for I obtained most protoxide of antimony when the evaporation was greatest.

These experiments serve however to demonstrate the true nature of the process. They show that instead of possessing the "uniformity" which we have been instructed to expect, uncertainty only occurs; and to such an extent that, contradictory and monstrous as it may appear, it is no less true than dangerous, that by this method we obtain products, the strength of which varies as widely as one from sixty, and may be of these or of any intermediate degree, without the existence of any obvious method of determining it, previously to exhibition. In fact the circumstance, that experiments similarly

conducted afford products, sometimes equally violent with the crocus antimonii, and at others as inert as the antimonium calcinatum, may at first appear to furnish an excuse for Dr. Powell's having represented these substances as synonymous: but had he, or had the College, known that the result of the process was liable to this extreme uncertainty, the promulgation of their directions must be deemed highly culpable; whilst the want of such knowledge is only to be attributed to unpardonable negligence.

When a process is so intirely devoid of every claim to adoption as that which is now under consideration, it is a question of curiosity only, how the less obvious difficulties attendant upon it are occasioned. It is evident that the superfluous nitric acid frequently peroxidizes a portion, and sometimes the whole, of the antimony; and it appears to me probable, but I state it only as a conjecture, that during the long time for which the solution is exposed to heat, some slight variation of temperature may cause the decomposition of the nitric acid to be effected either by the sulphur, or by the oxide of antimony, or by a portion of each. I have several times examined the solution, and have always detected some sulphuric acid in it. This however may occur in a considerable degree, and yet sufficient nitric acid may remain to peroxidize the antimony, for more oxigen is required to convert antimony into protoxide, than protoxide into peroxide, and I have already stated that to produce the first effect, one eighth of the nitric acid employed is sufficient.

Having already had frequent occasions to notice the state of various preparations obtained from Apothecaries' Hall, it may perhaps no longer excite surprise, that even those operators to whom Dr. Powell has confessed the

obligation of the College for their assistance in the contrivance of processes, should find it difficult to conduct them. In examining the oxide of antimony procured from this source, I have been anticipated by Mr. Stocker, who asserts [Pharm. Brit. p. 135.] that "in repeated trials made with the oxide obtained from Apothecaries' Hall, he has never yet succeeded in preparing tartarized antimony in its usual crystalline form, and possessing its ordinary activity." In corroboration of this statement I shall add that only one half of the oxide which I at first procured from thence, was dissolved when boiled in a solution of tartar; the solution, when evaporated, yielded crystals of supertartrate of potash, which did not contain a particle of oxide of antimony; and when treated with nitric acid its weight was not at all increased: in fact, instead of being the minor oxide, as Dr. Powell asserts, it was evidently altogether peroxide.

As however I had experienced so much uncertainty in this preparation, after a considerable time I again procured it from Apothecaries' Hall, and the difference between the two specimens is not less illustrative of the nature of this process than any circumstance hitherto mentioned. On this occasion I obtained an oxide, more than three parts of which were readily dissolved by four of tartar, and the solution afforded crystals of tartarized antimony. When treated with nitric acid, it increased about one seventeenth in weight; and it then became so nearly insoluble, that when two hundred parts of it were boiled in a solution of three hundred of tartar, only ten parts were dissolved. From these circumstances it is evident that it was truly the protoxide of antimony.

After what has been stated of the different medicinal powers belonging to preparations varying like these

procured from Apothecaries' Hall, I shall leave it to the reader to determine what effects may have been produced by practitioners who, relying upon the "uniformity" of this preparation, after having safely given ten grains of the first of these substances, have exhibited a similar quantity of the latter.

It is incredible that the College should have supposed that by this or any other process, an oxide could be obtained, at once capable of being formed into emetic tartar, and of being exhibited in so large a dose as ten grains. It must however be admitted that they intended, at least, to procure an oxide to be applied to the former purpose; and perhaps to that only, for the pulvis antimonialis, which is an effectual preparation of the protoxide of antimony, is retained in the Pharmacopæia.

Without giving an opinion as to the propriety of introducing any change into the process of making tartarized antimony, I have shewn the futility of the means adopted to remedy its defects, whether real or imaginary. A passage which I have quoted from Dr. Powell's translation seems intended to express an opinion, that the high temperature at which the crocus and glass of antimony are formed, either lessens or destroys their power: this opinion it is not necessary to refute; and I shall merely remark that it would be indeed extraordinary, if these preparations were deprived of their power by the very means requisite for their production.

Among other remarks upon the preparation of tartarized antimony, Dr. Powell observes, that "the former Pharmacopæia used the crocus antimonii, and others have preferred the antimonium vitrifactum, but it has been thought upon the whole, that the precipitated oxyd is the most uniform and definite, and yields whiter crystals." Upon the uniformity of this precipitated oxide any additional observations would be superfluous; and with respect to the whiteness here supposed to be obtained by the new process, I can state that I have procured crystals equally white when employing the crocus or glass of antimony. On examining the various experiments already related, it will appear that this is not the first instance in which imaginary objections have been made by the College, and I shall presently show that in this case they have been alleged when real ones have eluded their vigilance.

In the Pharmacopæia of 1787, tartarized antimony was directed to be prepared by boiling three parts of powdered crocus of antimony, for fifteen minutes, in a solution of four parts of tartar, and the filtered liquor was then to be suffered to crystallize. In order to make this preparation with crocus of antimony, I boiled one hundred parts of it, very finely powdered, in a solution of an equal weight of tartar, during a much longer time than is above directed; upon weighing the residuum it appeared that scarcely six parts of the crocus were dissolved. This experiment was repeated with very little variation in the result.

Although the crocus which I made use of appeared to be perfect, yet, finding so small a proportion of it dissolved, I thought it necessary to ascertain its purity. With this intention I boiled some of it in muriatic acid; scarcely an atom remained undissolved; and upon the addition of water to the solution a copious precipitation of submuriate of antimony ensued: this precipitate, after being washed, was dissolved in a solution of tartar, and gave crystals of tartarized antimony. These circumstances led me to suspect that the crocus, although finely powdered, was not sufficiently divided to admit of its

being dissolved by the tartar. I had recourse accordingly to some which had been levigated; but finding, on pouring an acid upon it, that considerable effervescence occurred, I suspected the presence of carbonate of lime: this was proved to be the case, by boiling it in dilute sulphuric acid, crystals of sulphate of lime being procured in considerable quantity. The respectability of the source from whence this levigated crocus was obtained satisfied me that this admixture was not fraudulent, but accidentally derived from the wearing of the stones employed in the levigation. The crocus being thus freed by sulphuric acid from the carbonate of lime, which would have prevented the formation of tartarized antimony, one hundred parts of it were boiled during some hours in a solution of an equal quantity of tartar: the undissolved portion weighed nearly 84 parts, consequently only about 16 parts were dissolved. I repeated this experiment, boiling the solution for a longer time, and on weighing the residuum it appeared that nearly 20 parts had been taken up by the tartar. It will be readily concluded that this proportion of oxide is not nearly sufficient to saturate the supertartrate; and still therefore apprehending that the cause of its not being dissolved was the want of more minute division, I procured a quantity of crocus which had undergone the process of elutriation; and I found, that by boiling one hundred parts of this in a solution of the same weight of tartar, the liquor assumed a deep green colour, and about 75 parts of the crocus were dissolved. This experiment was repeated with scarcely any variation; and crystals of tartarized antimony were obtained by evaporation. I now boiled one hundred parts of glass of antimony, powdered to the same degree of fineness as the crocus unsuccessfully used in the first of the experiments above related, in a solution of one hundred parts of tartar; the liquor assumed a deep green colour, which became gradually paler on exposure to the air, and at last yellow; depositing a precipitate of the same colour, after the tartarized antimony had been separated by crystallization. The insoluble residuum, after drying, weighed 22 parts; showing that 78 had been dissolved.

The cause of the green colour above mentioned, seems to be this: crocus and glass of antimony usually contain some oxide of iron. By the action of the tartar upon the fulphuretted antimony, sulphuretted hydrogen appears to be formed, which immediately acts upon the oxide of iron in the usual and well known manner. The probability of this opinion is shown by mixing a small quantity of a solution of peroxide of iron with one of antimony; for by the addition of sulphuretted hydrogen to the mixed solution, it assumes a green colour, very similar to that which I have described, and which totally prevents any indication of the presence of antimony. It is also worthy of remark that I have never observed this green colour, unless the tartar has taken up about three-fourths of its weight of the crocus or glass made use of; and I believe that it ought, under common circumstances, to be considered as indicating that a sufficient quantity of the metallic compound has been dissolved.

Bergmann has directed that the tartar should not be saturated with oxide of antimony; because in that case the solution, when set aside to crystallize, becomes gelatinous. Strictly speaking, however, it is not possible to saturate the supertartrate of potash; and even the crystals of tartarized antimony contain some acid in excess, as is sufficiently indicated by litmus paper. Bergmann does not seem to have been aware of the true nature of

this crystalline mass; and I believe it was first observed by Vauquelin, that tartar contains a considerable quantity of tartrate of lime. The fact is that acicular crystals of this substance, diverging from a common centre, form a great part of the salt obtained by crystallizing a solution of the tartarized antimony procured by either of the above processes, and an effect precisely similar is produced when a pure oxide of antimony is employed; the tartaric acid, which held the tartrate of lime in solution, becoming the solvent of the oxide of antimony. From these experiments it is apparent that crocus of antimony is extremely improper for the preparation of tartarized antimony; and I am apprehensive that much of the uncertainty attributed to this remedy, may have been occasioned by operators taking it for granted, that emetic tartar is always procurable by boiling powdered crocus of antimony in a solution of tartar.

The usual methods of preparing tartarized antimony, as well as that now adopted by the London College, are attended with sufficient inconvenience to authorize their rejection, provided a better process can be substituted; and the properties essential to such a process cannot be more clearly displayed than by exhibiting, at one view, the various objections which may be urged against the plans commonly recommended and pursued.

The method introduced into the late Pharmacopæia, is objectionable on the following accounts, viz. the proportion of crocus of antimony is too small; the ebullition is not continued for a sufficient length of time; the crocus is with difficulty reduced to a fine powder by the common means, and even in this state very little can be dissolved by the tartar; it becomes expensive by the tedious processes of levigation and elutriation; in addi-

with carbonate of lime, and the crystals of tartarized antimony are consequently contaminated even with a larger proportion of tartrate of lime than is furnished by the tartar.

To the use of glass of antimony nearly the same objections may be urged, excepting that it is much more easily powdered and more soluble than the crocus; consequently levigation and elutriation are unnecessary: it is however very expensive, and glass of lead, which it resembles, is sometimes fraudulently substituted for it.

The process now proposed by the London College, is absolutely inadmissible; the method of preparing the submuriate of antimony is inconvenient, unsafe, noxious, and tedious; its uncertainty without parallel, and its expense enormous; iron vessels cannot be employed in preparing it, and its quantity is too small in proportion to that of the tartar.

From Dr. Powell's observations, as well as from the name adopted by the College for the substance to be used in making tartarized antimony, it may be presumed that they intended to procure a mere oxide: by following their directions however, as already shown, a submuriate is formed; and this, for reasons presently to be assigned, is more convenient than the oxide.

Even when the most economical methods of procuring submuriate of antimony are pursued, its expense is great, otherwise I believe it ought to be preferred for making tartarized antimony. When a pure oxide of antimony, or one mixed with sulphuretted oxide is used, the tartrate of lime usually contained in tartar, as already shown, crystallizes with the tartarized antimony; but when submuriate is employed, the muriatic acid dislodged from

the oxide dissolves, and perhaps decomposes the tartrate of lime, and thus effectually prevents its crystallizing and contaminating the product: as, on this account, the submuriate may be occasionally preferred, I shall mention those which appear to be the best methods of preparing it. The inconveniences attending the process of decomposing sulphuret of antimony, induced me to try whether metallic antimony could not be advantageously substituted for it. By referring to p. 63 it will be seen that when using nitromuriatic acid, in the proportions directed by the Dublin College, each fluidounce decomposed such a proportion of sulphuret of antimony as yielded nearly two drachms of submuriate; but I found, to my surprize, that nitromuriatic acid thus prepared would scarcely dissolve more than one twelfth as much antimony as when the sulphuret was used; and that when the quantity of nitric acid was sufficiently increased to enable the muriatic to dissolve considerably more antimony, it was converted into peroxide, and precipitated during ebullition.

Submuriate of antimony may be obtained with much more convenience, and I believe with less expense, by using the crocus rather than the sulphuret of antimony. Let small pieces of the crocus be gradually added to heated muriatic acid, and when it appears to have taken up nearly as much as it is capable of dissolving, let the solution be boiled till the evolution of sulphuretted hydrogen gas has ceased. This ebullition is necessary for obtaining a white precipitate; it otherwise presents various shades of orange colour; and a precipitate of a brick-red colour was formed by decomposing a solution of crocus in muriatic acid which had not been heated.

I shall presently describe a preparation of antimony

which may be used for obtaining submuriate, more convenient, and not less economical, than the crocus.

It is evident from what I have now advanced, that the qualities requisite to form an eligible method of preparing tartarized antimony are these: the certainty of obtaining protoxide of antimony unmixed with peroxide or sulphuretted oxide, yet not absolutely pure, but mixed with some substance capable of preventing the crystallization of the tartrate of lime; moderate expense; and the possibility of using iron vessels both in preparing the oxide of antimony, and the tartarized antimony. It seemed to me probable that these requisites might be obtained by using subsulphate of antimony, prepared by boiling metallic antimony in sulphuric acid. I accordingly put 200 parts of the acid into an iron vessel, and added 100 of powdered antimony to it: the vessel was placed upon an open fire, under a chimney, and the mixture was stirred with an iron spatula as soon as it began to boil, and to emit sulphurous acid; and occasionally also till it became a dry mass: a greyish-coloured product was obtained, and was thrown into water, and washed till the uncombined sulphuric acid was removed: about 58 parts of antimony were oxidized, and the subsulphate, when decomposed by alcali, gave nearly 71 parts of oxide.

One hundred parts of subsulphate procured by this method, were boiled in an iron vessel with a solution of an equal weight of tartar: about 76 parts of the subsulphate were readily dissolved; and the solution, when filtered, afforded at the first crystallization rather more than 90 parts of crystals of tartarized antimony, which were perfectly white, and unmixed with any extraneous salt. After the separation of these crystals the solution was evaporated, and during this process, a very conside-

rable quantity of sulphate of lime was deposited, and separated before the crystallization of the remaining tartarized antimony occurred. The whole of the sulphate of lime was not however precipitated, and the crystals of emetic tartar were slightly incrusted with it: this was afterwards completely separated by redissolving the product, and suffering it again to crystallize. This process is so obvious as scarcely to require explanation. The sulphuric acid combined with the oxide of antimony in the subsulphate, decomposes the tartrate of lime, and forms sulphate of lime; and this being a salt of little solubility is precipitated.

As peroxide of antimony does not form a crystallizable salt with tartar, it may perhaps be safely concluded that every crystallized compound of tartar and oxide of antimony is emetic; but since, in a matter of so much importance, it is improper to confide in any inference however plausible, I requested Mr. Stocker and Mr. W. Brande to ascertain the efficacy of the tartarized antimony procured by employing the subsulphate: their report of its activity was such as fully to justify me in recommending the method as far as efficiency is concerned.

This process is not tedious, difficult, uncertain, or unsafe; and it remains only to be shown that it is economical, to prove that it is more advantageous in every respect than the methods for which I propose to substitute it: this I shall do by presenting a comparison of the cost of the oxides obtained by this process and by the ordinary methods.

tapemono

One part of glass of antimony			 costs		48
do.	CLE IN	10			22
metallic do.	0 01				21
sulphuret of do.	4330	·V			12
sulphuric acid	1977	•			4
muriatic do.		770			5
nitric do.	. View				20

Glass of antimony is composed of about 8 parts of protoxide, I of sulphuretted oxide, and I of silica, on which account, 8-10ths only being convertible into tartarized antimony, its cost must be estimated at 60; and one fifth part of the crocus being sulphuretted oxide, its cost is raised to 27. I have shewn that 58 parts of antimony which cost 1218, and 200 of sulphuric acid at an expense of 800, produce 71 parts of oxide, = 28.5 cost of one part of oxide. It appears at p. 63 that two parts of sulphuret of antimony give 1.75 part of submuriate, containing by estimation about 1.6 of oxide of antimony: to produce this the London College employ, of sulphuret of antimony, 2 parts, cost 24

muriatic acid, 12.16 parts, . . . 60.8 nitric acid, 1.42 parts, . . . 28.4

113.2 total

cost of obtaining 1.6 part of the oxide, = 70.75 for one part. I have estimated the muriatic and nitric acids at the rate of common preparations, as they answer equally well for that purpose with the purer and more expensive Collegiate acids, and they are such as would certainly be employed by those who prepare tartarized antimony in large quantities. Submuriate of antimony may, however, be much more economically prepared by using the

nitromuriatic acid directed by the Dublin College, and by adding to it as much sulphuret of antimony as it is capable of decomposing: I have stated, at p. 63, that only 14 drachms of submuriate are procured by the Dublin process, whilst the proportion of acid which is directed will decompose as much sulphuret as yields 22 drachms of submuriate. By this method of decomposing sulphuret of antimony by nitromuriatic acid, the cost of obtaining oxide will be reduced from 70.75 to about 41.6.

The expense of procuring oxide of antimony by the different methods will be nearly in the following proportions: From

In these calculations I have not comprehended the expense of time and fuel consumed in preparing the subsulphate, and submuriate of antimony; on this account the difference between the cost of the glass of antimony which it is requisite only to pulverize, and that of the other substances, is not so great as it appears to be. The expense of levigating and elutriating the crocus of antimony is considerably greater than that of time and fuel employed in forming the subsulphate; but it is less than that of preparing the submuriate by the process of the London College; which is indeed so tedious, and consequently so expensive, that it is easier to obtain, in the same space of time, ten parts of subsulphate by the method which I have proposed, than one part of submuriate by this process. That of the Dublin College re-

quires much less attention, for the nitromuriatic acid directed by them to be used is not so violent in its action as that of the London Pharmacopæia, consequently more sulphuret of antimony may be added to it at once.

The best method of preparing submuriate of antimony is to dissolve the subsulphate in muriatic acid; it is much more easily taken up than the crocus, and the acid does not require ebullition, as is the case when the crocus is employed after being saturated with it.

There is one important point connected with the preparation of tartarized antimony, which has not excited sufficient attention; I mean proportioning the antimonial preparations according to the quantity of oxide convertible into tartarized antimony which they contain.

To determine the proportion of oxide of antimony which tartar is capable of dissolving, I decomposed some submuriate by heating it in a dilute solution of subcarbonate of potash. The muriatic acid was perfectly separated by the alcali; for when the oxide was dissolved in tartar, nitrate of silver being added to the solution, no precipitation occurred. One hundred parts of this oxide were boiled in a solution of an equal weight of tartar; the oxide undissolved, after being washed and dried, weighed 30 parts, consequently 70 parts were dissolved: this experiment was many times repeated without variation in the result. When however 70 parts of oxide are boiled in a solution of 100 parts of tartar, the whole of the oxide is not taken up, and I have found by repeated experiments that only about 66 parts are dissolved. It is not easy to account satisfactorily for this circumstance. The practical inference to be drawn from this experiment is, that more oxide of antimony should be used than the tartar is capable of dissolving: no inconvenience is occasioned by the undissoved portion, and when subsulphate or submuriate is employed, the residuum is equally proper for use as at first, and in the same proportions; the case is however obviously and widely different with the residuum of the glass or crocus of antimony.

When equal weights of tartar and any of the following substances are boiled together in water, the proportions annexed to each are dissolved:

oxide of antimo	ny				70
glass of	do.				78
crocus of	do.			. nearly	75
submuriate of	do.			from 74 to	77
subsulphate of	do.			from 74 to	78

The apparently greater solubility of the oxide of the glass and crocus than of the pure oxide, depends probably on the evolution of sulphuretted hydrogen and the solution of sulphuretted oxide; and it is difficult to discover whether the solution of the oxide is prevented by this circumstance, and to what degree: this uncertainty is an additional objection to the use of these preparations. The greater solubility of the submuriate and subsulphate evidently depends, as well as the variations in their solubility, upon the quantity of acid which they contain. When submuriate has been washed with cold water till it ceases to redden litmus paper, boiling water will produce this effect; it is however improper to use hot water in washing it, because it is to a certain extent soluble therein.

Although, for the reasons I have stated, the use of the subsulphate of antimony appears to me much more eligible, yet others may prefer the use of the glass or crocus: in employing them, it should be remembered that the glass ought to be very finely powdered, or if levigated, as some advise, that it should be freed from carbonate of lime by boiling in dilute sulphuric acid; and the levigated crocus should undergo the same process either before or after the elutriation: and, lastly, when either of these substances is employed, I would recommend the addition of a small quantity of sulphuric acid, to decompose the tartrate of lime, in the same manner as it is effected when the subsulphate of antimony is made use of.

The following are the proportions of the annexed substances which it appears to me proper to employ with 100 parts of tartar:

glass of anti	antimony		110
crocus of	do.		110
oxide of	do.	TO	80
submuriate of	do.		90
subsulphate of	do.		90

Although tartarized antimony is extensively employed, there are but few apothecaries who prepare it: consequently it is mostly purchased of those persons who make it in considerable quantity; and by them it is usually offered for sale in the state of powder, in order to prevent that detection of its imperfections, which is easy when the salt retains its crystalline form. It is readily procured in crystals of considerable size, and therefore those who purchase it ought to insist upon having it in crystals sufficiently large to be submitted to examination; either by ascertaining their figure, which I shall presently describe, or by the easier means which are in this instance afforded by chemistry.

From every fresh quantity of crystals received, a few ought to be promiscuously taken and washed with a small quantity of water; they should then be separately introduced into dilute solutions of the potassæ sulphuretum. If, on standing for a short time, an orange coloured precipitate occur in considerable quantity in each solution, the salt may be safely regarded as being perfect. If, at any time, crystals should not be procurable, the powder ought to be similarly examined; but as it may consist of a mixture of tartarized antimony and tartar, its nature should be ascertained by attempting to prepare the liquor antimonii tartarizati with a portion of it. If the salt do not readily and totally dissolve in the water, and form a perfectly clear solution previous to, as well as after, the addition of the wine, it ought to be rejected, as containing tartar uncombined perhaps with any oxide of antimony, or at least with a proper quantity of it.

The primitive crystal of this salt is a rectangular octohedron, the planes of which meet at the summit at an angle of about 63°; and like other substances which possess a similar primitive form, it admits of the tetrahedron as one of its varieties. It is usually difficult to trace the form of the crystals, on account of the numerous variations to which the octohedron and tetrahedron are subject; and more especially because the crystals are very commonly incomplete.

# LIQUOR ANTIMONII TARTARIZATI.

In the late Pharmacopæia the vinum antimonii tartarizati was directed to be prepared by dissolving two scruples of tartarized antimony in a mixture of ten fluidounces of water and wine. The College have now reduced the

tartarized antimony to one scruple, and this important alteration is not only unnoticed by Dr. Powell, but he even states that "this preparation resembles the former vinum antimonii tartarizati." There is not the slightest difficulty in making the former preparation; the change introduced must therefore be considered as originating in mere frivolity, and it may occasion serious disappointment to those practitioners who are not aware that it is requisite for the safety of their patients, to examine how many alterations have been made, without even the hope of improvement. In order to obviate as much as possible the difficulty which this and similar variations have occasioned in compounding, I employ the old or new preparation according to the name used by the practitioner in prescribing: this method, as I have stated on a former occasion, may not in every instance prove effectual, but it is the only one which I have been able to contrive.

### PULVIS ANTIMONIALIS.

In the Pharmacopæia of 1787, equal parts of sulphuret of antimony and hartshorn were ordered to be employed in making this preparation; in the present Pharmacopæia the sulphuret of antimony is reduced to one half its former quantity, and the reason which Dr. Powell has stated for this great alteration appears to be singularly unworthy of avowal: "This preparation was introduced into the last Pharmacopæia, as a substitute for a medicine of extensive celebrity, Dr. James's powder; to which however the present form more nearly assimilates in its dose, and it is more manageable in its administra-

tion, by the reduction of the proportion of antimony to

Thus, if this statement be authentic, a preparation of more than twenty years standing has been deprived by the Royal College of Physicians, of one half of its power, merely that it may more nearly resemble a quack medicine, the use of which it has almost superseded! A better reason might have been assigned; for I have been informed, by unquestionable authority, that the change now directed has been long adopted on account of the impossibility of obtaining a white product by the former process.

#### ARGENTI NITRAS.

One ounce of silver is directed to be dissolved in one fluidounce and a half of nitric acid previously diluted with water. This quantity of acid is much larger than is necessary: Dr. Powell observes that "nitric acid dissolves half its weight of silver," and the proportions in the Pharmacopæia are nearly such as they ought to be, if this statement were correct. I find however that 100 parts of nitric acid dissolve about 117.35 parts of silver; consequently one fluidounce and a half, weighing 1022.62 grains, the quantity ordered for the solution of 480 grains of the metal, are capable of dissolving 1200 grains.

#### ARSENICI OXYDUM PRÆPARATUM.

Arsenic is now introduced into the Pharmacopæia. The resublimation of it which is directed, is however useless, for the "shining semi-vitreous lumps" mentioned

by Dr. Powell, are as pure as oxide of arsenic can be rendered by sublimation, or probably by any other method. From the process which the College have recommended, it seems as if they imagined that the imperfection of an operation could be obviated by repetition.

## LIQUOR ARSENICALIS.

The appellation bestowed upon this preparation is remarkably indefinite; and it would have been equally consistent with the chemical nomenclature sometimes adopted by the College, to have called the liquor ammoniæ acetatis, by the name of liquor ammoniacalis.

The formula introduced into the Pharmacopæia seems intended to resemble that proposed by Dr. Fowler, and since very generally adopted.

I have already had occasion to notice one absurd application of the potassæ subcarbonas e tartaro; but employing it for a solvent of oxide of arsenic, is if possible, a more glaring instance of useless refinement. Do the College imagine that when the common subcarbonate of potash is used, the impurities obstruct its action upon the oxide? or that the impurities are dangerous in the same doses in which arsenic is not merely safe but salutary?

The mode of preparing this extremely active medicine is very loosely and imperfectly described by the College, and rendered yet worse by the translator. The oxide of arsenic and subcarbonate of potash are to be boiled together in a pint of water until the arsenic is dissolved; four fluidrachms of compound spirit of lavender are then to be added to this solution, and lastly a quantity of water, sufficient to occupy with it a pint measure.

Now I have found that the arsenic has been completely dissolved before even two fluidrachms of water were evaporated; and consequently, if four fluidrachms of spirit of lavender had been put to the solution, the intended quantity would have been exceeded, without the addition of any water. A certain and rational direction would have been, that the ebullition should be continued until a specified portion of water was evaporated.

Dr. Powell has rendered the directions very incorrectly in both editions of his translation. In the first he says, " add as much water as may exactly fill a pint measure;" in the second edition it is altered to "add as much more distilled water as may be requisite to make up a pint measure:" but as it is not stated in either version that it is the arsenical solution and the water to be added to it, which are together to "fill" or "make up a pint measure," the words made use of are merely circuitous expressions for-add a pint of water to the solution. It is evident therefore, that by observing these injunctions, the solution of arsenic is reduced to about one half of its intended strength; but these very errors have probably been productive of safety, by preventing the fatal effects of some most gross and unpardonable blunders, which Dr. Powell has committed with respect to the doses of "arsenic, the most virulent of the mineral poisons." He states, and, if the solution be prepared according to the directions of the text, states almost correctly, that " each ounce," or, as he ought to have said, each fluidounce, "contains four grains of oxide of arsenic;" and then adds that "each drachm," being one eighth of an ounce, "contains one eighth of a grain," instead of one half of a grain of oxide. It is nauseating again to notice this monstrous error, which has been so much animad

yerted upon: it is however requisite, because in the prodigious list of errata, of which list, I know not whether to say fortunately or unfortunately, copies were with difficulty to be procured, this mis-statement, although noticed, is not rectified, but merely diminished; the words "fluid half drachm" being directed to be substituted for drachm. It is however evident that a fluid-half-drachm instead of containing, according to this new reading, one eighth of a grain, contains one fourth.

## CUPRUM AMMONIATUM.

This preparation is now first introduced into the London Pharmacopæia, and the method of preparing it, which is copied from the Edinburgh Pharmacopæia, answers perfectly well.

# LIQUOR CUPRI AMMONIATI.

The process inserted in the Pharmacopæia of 1787 for the preparation of the aqua cupri ammoniati, is extremely inelegant. A mixed solution of lime and muriate of ammonia was to be saturated with copper by standing in a vessel of that metal. This method might have been easily improved; and the present formula, until it is tried, has every appearance of being preferable to the former. One drachm of the cuprum ammoniatum is to be dissolved in a pint of water: but the fact is, that owing to the large quantity of water employed, about one half of the cuprum ammoniatum is decomposed, and the oxide of copper which it contained is precipitated: the solution is therefore of only half the intended strength. One fourth

of the quantity of water now used, or even less, is sufficient to dissolve one drachm of the cuprum ammoniatum.

### FERRUM AMMONIATUM.

It appears probable that the peroxide of iron directed to be used in the Edinburgh Pharmacopæia, is preferable to the metallic iron ordered in the late Pharmacopæia for the preparation of ferrum ammoniatum. If however the College had tried the carbonate of iron for this purpose, its use would not have been directed; for in proportion as it contains carbonic acid, carbonate of ammonia is formed, instead of ferrum ammoniatum.

#### FERRI CARBONAS.

In the Pharmacopæia of 1787, peroxide of iron was prepared, under the name of ferri rubigo, by exposing iron filings to the action of air and water until converted into red oxide. This method is imperfect; and the introduction of a precipitated peroxide is certainly an improvement. This preparation has been for a considerable time employed, under the name of ferrum præcipitatum: from the appellation given to it by the College, it appears that they intended it to consist of oxide of iron combined with carbonic acid. I am not however aware that carbonic acid has been supposed to increase the efficacy of the oxide, and unless this were decidedly the case, it would have been much better to have directed the preparation of a simple oxide.

This substance is directed to be prepared by mixing hot

solutions of subcarbonate of soda and sulphate of iron; and the precipitate obtained by their mutual decomposition is to be washed with hot water and dried. This method is borrowed from the Dublin Pharmacopæia, and when accurately observed, an oxide of iron is procured which certainly contains a considerable quantity of carbonic acid: it appeared however to me probable, that the proportion was not sufficiently large to intitle the preparation to the name of carbonate of iron. To determine this I made the following experiment. A solution of one hundred parts of sulphate of iron was decomposed as proposed in the Pharmacopæia, and the precipitate, after being washed with hot water, on being dissolved, whilst moist, in muriatic acid, gave out 5 parts of carbonic acid: now Kirwan states that sulphate of iron contains 28 per cent. of oxide; but this quantity can only be obtained by converting the oxide into peroxide; and as 100 parts of this contain 66.6 of protoxide, 28 will give 18.6 as the quantity of protoxide in 100 parts of sulphate of iron. According then to the experiment just stated, 18.6 of protoxide of iron are capable of combining with 5 of carbonic acid; and consequently, 100 parts of dry precipitate ought to contain 21 of carbonic acid; but after repeated trials I have never been able to obtain a carbonated oxide which gave out more than 14.5 per cent. of carbonic acid, although dried by the heat of steam within twelve hours of the time of its precipitation.

It is therefore evident that part of the oxide is not combined with any notable quantity of carbonic acid; and consequently, that the precipitate is either a mixture of black and red oxide and of carbonate or subcarbonate of iron, or altogether a subcarbonate. The latter supposition is less probable than the former, because the

appearance of a solution of the precipitate in muriatic acid, denotes that a part of it was in the state of peroxide; and I have in vain endeavoured to procure a solid compound of peroxide of iron and carbonic acid, by decomposing oxisulphate of iron with solution of carbonate or subcarbonate of soda; the precipitate obtained, when dried by exposure to the air, never giving out any carbonic acid by solution in muriatic acid. These statements confirm the suspicion which I have already expressed, that this preparation is not a perfect carbonate of iron; and consequently, it is improperly so called.

The process now introduced into the Pharmacopæia is very objectionable on several accounts; for although the proportions of the metallic and alcaline salts be exactly observed, yet very slight deviations from the proceeding directed by the College, occasion so much variation in the appearance of the products, as to render it very difficult to ascertain their identity. This appears by the following table of experiments, in which the proportions of subcarbonate of soda and sulphate of iron recommended by the College, were employed:

	Hot Water	Hot Water	Steam	14.5	Chocol.brown
			Expes. to the air	14.5	Yellowish br.
ed in	P	Cold Water	Steam b	1.5	Orange brown
ipitate	Cold Water	Hot Water	p	8.0 olo	Purplish br.
Preci		Cold Water	nic s	1.0	Reddish br.
-	Single and a	l	Expos, to the air	none	Ochre yellow
.48	Water kept near	212° for one hour	Steam	1.5	Blackish br.

In this statement it appears that very obvious differences are effected, not only in the aspect of the products, but also in the degree of their oxidizement, and the quantity of carbonic acid which they contain; and that these effects

are produced by slight variations in the temperature of the water employed, and in the method of drying the precipitates.

The variation in the colour of the precipitates of peroxide of iron does not appear to indicate any essential difference in their nature. When the oxigen is rapidly absorbed the product is of a deep red; when slowly, it is of a yellow colour; protoxide of iron is, as is well known, black, whilst its carbonate appears to be brown; and the precipitates which are above described evidently consist of these oxides and carbonate of iron, mixed in different proportions.

To discover the cause of the different effects produced by employing hot and cold water, I separated by filtration, the oxide procured by the latter method from the fluid in which it was precipitated, and on adding hot water to this fluid, precipitation of carbonate of iron immediately ensued; the carbonic acid which held it in solution being expelled by the heat. It is evident therefore, that the solubility of carbonate of iron in cold water, is the reason why the precipitates obtained when using cold solutions, contain so little of this compound; whereas when a moderate degree of heat is applied, that portion of the carbonic acid which held the carbonate in solution is expelled, and the carbonate is consequently precipitated: if however a considerable degree of heat be employed for a length of time, as in the seventh experiment, the carbonate itself is decomposed, and the precipitate consists, for the most part, of black oxide of iron.

It certainly would be possible to avoid in a very considerable degree the varying effects produced by the Collegiate process, by strictly observing the minutiæ of it; but the inconvenience which I have described when treating of ferrum ammoniatum, would be greater than when the precipitates which contain less carbonic acid are employed. Another defect of the present process is its costliness, 10 parts of subcarbonate of soda being directed for the decomposition of 8 parts of sulphate of iron: now I find that 5 parts of subcarbonate of potash are capable of producing a similar effect, and at less than one third of the expense; the precipitates obtained are also much less subject to vary by slight changes of temperature than those procured by subcarbonate of soda.

It is evidently proper to employ hot water in every stage of the process to prevent the solution and waste of carbonate of iron; and although, when subcarbonate of potash is used instead of subcarbonate of soda, and this method is also adopted, the precipitate contains only half the quantity of carbonic acid, yet 7 per cent. of carbonic acid is sufficient to occasion considerable loss in preparing the ferrum ammoniatum.

The last described method appears to me to be preferable to any of the others; yet it is not free from the inconvenience of containing some carbonic acid, and perhaps in making the ferrum ammoniatum it would be better to employ the red oxide of iron, as directed in the Edinburgh Pharmacopæia.

I have already described seven varieties of precipitates

obtained by using the materials directed in the London Pharmacopæia; and the preparation procured from Apothecaries' Hall is different from every one of them: it is of a brick red colour, and contains 1.5 per cent. of carbonic acid, resembling most the second precipitate above described, obtained by using subcarbonate of potash.

### FERRUM TARTARIZATUM.

This preparation of iron has been long in partial use in a very imperfect state; and its properties do not seem to have been investigated with precision adequate to the importance of the subject. In the Pharmacopæia of 1787, one pound of iron filings, and two pounds of tartar in powder, previously mixed, were directed to be moistened with a pint of water; and this mixture, after being exposed during eight days to the air, was to be dried by the heat of a water bath, and pulverized. Thus prepared it is usually of a light green colour, readily attracted by the magnet, unalterable by exposure to the air, and with difficulty soluble in water; and that which I obtained from Apothecaries' Hall contained 20 per cent. of metallic iron intirely unchanged by digestion with the tartar. In this state it is evidently a mere mixture of iron with supertartrate of potash slightly coloured by oxide of iron, and its efficacy is probably dependant upon the metallic iron which it contains.

In order to obviate the imperfections which I have now described, the College have properly ordered the digestion to be continued for a greater length of time than before; but the exsiccation of the mass, directed to be performed before the saturation is completed, is not only

unnecessary, but as I shall hereafter show, prevents the preparation from being exhibited in the most eligible mode.

It is of considerable importance to attend to the nature of the iron employed in this preparation. I mixed 64 parts of tartar with 32 of filings of soft iron, and of these 17 parts remained unacted upon after long digestion; but I found, when I used the filings commonly employed, and which probably were steel, that 19 parts were unacted upon by the tartar.

To prepare this compound properly, water should be occasionally added to the mass during the action of the tartar upon the iron; and the digestion should be continued until it appears, by the test of litmus paper, that the acid is perfectly saturated with iron.

This preparation has been very commonly prescribed to be used in the state of powder, but this cannot be conveniently done when the more perfect salt obtained by long digestion is employed, because it attracts moisture from the atmosphere. The form in which I have recommended it to be exhibited, and which has met with the approbation of practitioners of eminence, is a solution: but if the mass has been dried, even with the heat of steam or a water bath, it is difficult to obtain a perfect solution: for by the action of the heat it becomes exceedingly tenacious, and is converted into a substance very much resembling gum; and in this state it is almost impossible to obtain a clear solution, either by subsidence or filtration.

I have already stated that 64 parts of tartar are capable of dissolving 15 parts of iron, and these are converted into nearly 22 parts of peroxide. To this, I add 7 times its weight of water; which easily dissolves the tartarized

iron when triturated, and the fluid readily passes through the filter. By these means a solution is obtained which contains one eighth part of its weight of tartarized iron; or about 3.3 per cent. of peroxide: therefore each fluidounce will be found to contain very nearly 16 grains of oxide.

The advantage of exhibiting this preparation in the state of solution, arises from the taste of iron being scarcely perceptible when the acid is perfectly saturated; for it is a circumstance worthy of notice, that when there is acid in excess, the taste of iron is much more easily detected. It follows therefore from what I have now stated, and experience corroborates the inference, that this solution may be exhibited with success to those persons to whom the common solutions of iron are nauseous.

The solution prepared as above described is of a deep greenish brown colour, and it remains for a great length of time without undergoing any change; it is indeed true that soon after it has been prepared considerable precipitation sometimes occurs, but this precipitate is not tartarized iron. I washed a quantity of it upon a filter, until the distilled water used in the operation came through nearly colourless, and the precipitate retained only a slight tint of yellow: this circumstance is of itself sufficient to prove that it could not be tartarized iron. Some of it was then boiled in distilled water, and on adding oxalate of ammonia to the filtered solution, precipitation immediately ensued, showing that the precipitate consisted of tartrate of lime, which as already noticed is usually contained in tartar.

Tartarized iron when in the perfect state I have decribed, is of a dark greenish brown colour, and attracts,

as already mentioned, moisture from the atmosphere, but does not deliquesce. From an aqueous solution it is precipitated by alcohol, and it is decomposed by lime water; tincture of galls renders it black, but prussiate of potash acts very slowly and imperfectly upon it; in the course of some days a greyish coloured precipitate is however obtained, but not at all resembling prussiate of iron as usually prepared; if indeed the solution contain excess of acid it becomes quickly green on the addition of this reagent, and after some days blue prussiate of iron is deposited; potash and soda decompose this solution slowly unless heated, but in this case peroxide of iron is readily precipitated, as also by their subcarbonates, which when cold, produce no change; neither ammonia nor its subcarbonate decomposes the solution whether hot or cold, and it does not crystallize as asserted by Dr. Powell: it is indeed possible that crystals of tartar slightly coloured by oxide of iron may be obtained, by dissolving the imperfect salt already noticed.

# LIQUOR FERRI ALCALINI.

"This preparation," observes Dr. Powell, "was first described by Stahl (Opusc. Phy. Chem. Med. Hal. 1715.) and called Tinctura martis alcalina, it is now first introduced into the Pharmacopæia, as affording a combination of iron, distinct from any other, and often applicable to practice;" but as it is afterwards stated that its "chemical composition has not been very exactly ascertained," the first of the reasons alleged for employing it, is rather too venturously advanced; and the negative praise contained

in the latter, would apply to iron in every state, however tortured by ingenuity or by ignorance.

It is frequently as instructive to trace the wanderings of error as the progress of truth, and the article now presenting itself for consideration exhibits an accumulation of pernicious effects, arising from that deplorable deficiency of experiment on the part of the College, to which I have so frequently adverted: that this deficiency, unfortunately for themselves and for the community, did exist, I shall on this occasion demonstrate, by displaying the formulæ for making this preparation inserted in the Specimens issued by the College; and by comparing them with each other, and with that which was eventually adopted, and now constitutes a part of the Pharmacopoeia.

In Dr. Powell's preface to his translation, it appears that the committee appointed by the College commenced their meetings in January, 1806; and that after a deliberation of more than two years, a "Specimen" of the progress they had made towards completing the important object entrusted to their care, was printed with the sanction of the College.

In this "Specimen" the following proportions were assigned for preparing the

"Solution of alkalized iron.

"Take of iron filings, four drachms,
nitric acid, two [fluid]ounces
distilled water eight [fluid]ounces.

"Having diluted the acid with the water, add it to the iron filings, and after the effervescence has ceased, strain it. To the strained solution add gradually, solution of subcarbonate of potash, and shake it until the powder at first

precipitated is again dissolved. Set it aside that the nitre may crystallize, and pour off the solution."

In the "Specimen alterum" the following directions

were proposed:

"Take of iron, four drachms,

nitric acid, two [fluid]ounces,

distilled water, eight [fluid]ounces,

solution of subcarbonate of potash, one[fluid]ounce.

"Pour the acid and water, previously mixed, upon the iron; and when bubbles cease to arise strain the solution. To this add gradually the solution of subcarbonate of potash, until the powder at first precipitated, is again dissolved. Set it aside, that the nitrate of potash may crystallize, and pour off the clear solution."

The formula adopted is the following:

"Solution of alkaline iron.

"Take of iron, two drachms and a half.

nitric acid, two fluidounces,

distilled water, six fluidounces,

solution of subcarbonate of potash, six fluidounces.

"Having mixed the acid and water, pour them upon the iron, and when the effervescence has ceased, pour off the clear acid solution: add this gradually, and at intervals, to the solution of subcarbonate of potash, occasionally shaking it, until it has assumed a deep brown red colour, and no further effervescence takes place. Lastly, set it by for six hours, and pour off the clear solution."

As the quantity of solution of alcali necessary to be employed in the process proposed in the first Specimen is not mentioned, it was of course intended to be left to the operator to employ a sufficient portion of it: this method

is certainly very improper, because it would be impossible for any practitioner to discover what measure of fluid contained the quantity of oxide of iron which he wished to exhibit; and consequently, without previous inquiry or experiment, he could not ascertain the dose of the preparation.

This is not the only objection to the process, for experiment shows that it would be equally impossible for a practitioner to ascertain the quantity of the iron, as that of the solution containing it; for when a small portion of the diluted acid is poured upon the filings, the action is so rapid, owing to the divided state of the iron, that a portion of it is precipitated in the form of peroxide, and is not soluble when the remaining acid is added. If on the other hand the whole of the acid be added at once the action is still more violent, and the solution is expelled from the vessel. Taking it then for granted that these circumstances were made known to the committee, by some person who had tried the experiment, let us next examine how it was proposed to remedy these defects.

In the "Specimen alterum" the iron is not directed to be in the state of filings, and the quantities of it, and of the nitric acid and water, remain as in the "Specimen," but a definitive quantity of the subcarbonate of potash is proposed; the quantity assigned could not however have been recommended as sufficient, in consequence of experimental decision; but the committee imagined no doubt that it was capable of effecting the intended purpose. They must therefore have supposed that the alcali contained in less than six drachms of subcarbonate of potash was so much more than was necessary to saturate the acid undecomposed by the action of the metal, as that the remainder

would be sufficient to dissolve nearly six drachms of peroxide of iron. It is scarcely requisite to mention that this proportion of alcali is absolutely inadequate to answer even the first purpose, that of saturating the acid, and the College afterwards increased it to about six times the quantity; evidently, as I shall hereafter show, without knowing the extent of the deficiency.

I shall now examine the formula which was at length deemed sufficiently perfect for adoption. In this the quantity of iron is reduced to five-eighths of that directed in both the Specimens; and Dr. Powell alleges that "the directions given by Stahl, are by no means certain in their effects, and seem especially erroneous in ordering the complete saturation of the acid with the iron;" consistently with this opinion, he has advised that the solution should be "not nearly saturated and very acid," and he observes that "in this state it has not the reddish yellow colour of a saturated solution, but is clear and slightly greenish. This is intended to be effected by the directions given: but if by accident the solution should go further, the proper colour is immediately restored by the addition of a small quantity of acid." Now what is meant by the solution "going further," is not very evident: according to the directions in the original, the acid and iron are to be suffered to act upon each other until the effervescence ceases, and the effervescence does not cease until the iron is dissolved; consequently in the opinion of the College, the solution cannot go too far. If however, Dr. Powell had determined by experiment, that the proportion of iron dissolved was too large, it would have been much more definite to have employed less of it, than to have directed that the solution should be regulated according to the very uncertain

indications afforded by the variations of its colour; and it is an obvious omission not to have stated that the alcali should be increased to saturate the acid employed for restoring the colour of the solution. Thus then if Dr. Powell's advice be followed, the unlimited addition of acid and alcali would destroy the seeming precision of this process, and render the dose of the solution as uncertain as that obtained by the formula proposed in the first specimen, and very properly rejected from the second, although its place was supplied by a worse.

The fact however is, that it is not requisite that the solution should be extremely acid, but even if it were so, the present proportions of acid and iron may be employed without any addition of the former; for I found that the acid used to dissolve two and a half drachms of iron was capable of dissolving more than five, without the application of heat; and the solution thus obtained, although so nearly saturated as to excite but little effervescence when added to the solution of subcarbonate of potash, answered perfectly well for making this preparation.

The intention of employing a very acid solution seems to be to decompose some of the subcarbonate of potash by the excess of acid, in order that the carbonic acid evolved may assist the remainder in dissolving the oxide of iron; the experiment above related shows however, that the precaution is useless, and consequently that it occasions a waste both of acid and alcali.

Dr. Powell observes that the preparation of this medicine "will succeed by the gradual addition of either solution to the other, but it has appeared to me to be more certain when made according to the directions given in the text, and by shaking the mixture after each addition of the acid solution to the alcaline one." When the

proportions of the solutions are properly adjusted, the preparation may certainly be effected by the addition of either fluid to the other; Dr. Powell however justly prefers adding the acid solution to the alcaline one: but it happens very unfortunately, that neither method will succeed when the solutions proposed by the College are employed, because the proportion of alcali is too small; and I find it necessary to use about one twelfth more than is directed. The College seem indeed to have been aware that the method which they have introduced into the Pharmacopæia was imperfect; for Dr. Powell observes that "the proportions" of acid and alcali "are pretty nearly as there given, but they require to be checked by occasional examinations, particularly by the taste, which ought to have a small predominance of alkalescence in it." Now the fact is, that the proportion of alcali, although too small, as already stated, to dissolve the oxide of iron, is more than sufficient to saturate the acid; consequently the solution may have an alcaline taste, and may be, as it actually is, very imperfect: indeed when the alcaline solution is added to the acid almost the whole of the oxide of iron is precipitated.

Now had experimental decision guided the proceedings of the College, much inconvenience would have been spared; they would not in both Specimens have directed the alcaline solution to be added to the acid one, nor have advised filings of iron to be used instead of a "lump," to which Dr. Powell has given well merited preference; they would not have recommended for the saturation of the nitric acid an insufficient quantity of subcarbonate of potash; nor have directed about an equal weight of peroxide of iron to be dissolved by the imaginary overplus. Experiment would moreover have

shown Dr. Powell, that it requires much more alcal; to dissolve the oxide of iron when it is added to the acid, than when the contrary method is used; consequently that although the solution may be effected by either mode of mixing, yet the same proportion of alcali is not applicable to both. It would also have been found that it is not requisite that the solution should be very acid; and that if it were needful, it is not necessary to add more acid to a fluid containing scarcely half the quantity of iron which it is capable of dissolving: and lastly, a few hours of experiment instead of as many years of speculation, would have prevented the recommendation of one improper and two impracticable processes, for making what is strangely denominated, in defiance of chemical propriety, solution of alcaline iron.

The most remarkable characteristic of this solution, and which really distinguishes it from all other preparations of iron with which I am acquainted, is it's decomposition by the addition of water. When five parts of water are added to one part of this solution, the oxide of iron is almost entirely precipitated in a few minutes.

When it is considered that this medicine is now introduced into the Pharmacopæia, it must appear singular that so unusual a property as that which I have just described is not noticed by Dr. Powell; for as there is no circumstance in the nature of this solution which can a priori lead to a suspicion that water decomposes it, the unexpected occurrence of a precipitate where a solution is directed, may induce practitioners to suspect that the medicine is improperly prepared.

Mr. Stocker in his observations upon this preparation (Pharm. Off. Brit. p. 230) remarks that "it is no doubt

intended, from the nature and proportions of the ingredients employed in the foregoing process, that the preparation resulting from it should consist chiefly of subcarbonate of iron held in solution by the subcarbonate of potash; but from the instructions given to the operator, and the circumstances by which he is to determine in what proportion the materials are to be used, he must necessarily be embarrassed. For he is told that the acid solution of iron is to be added in divided portions to the alcaline liquor, not only until the mixture becomes of a brownish red colour, but until there is no longer any extrication of gas, an effect which must continue to take place even on the addition of the least portion of it." It must be admitted that Dr. Powell's translation will bear this construction, but the true meaning of the original appears to be this: Shake the alcaline solution after each addition of the acid until gas ceases to be evolved.

Being desirous of examining this medicine, prepared with the advantages enjoyed by those operators, from "most important intercourse" with whom this process probably emanated, I applied for it at Apothecaries' Hall. The solution which I obtained possessed properties diametrically opposite to those belonging to an alcaline solution of iron, as will appear by the following statement of the effects produced by reagents upon each.

r may be conveniently	Alcaline Solu- tion of Iron.	Solution from Apothecaries' Hall,
Turmeric paper	Reddened	. No effect
Litmus paper	No effect	. Reddened
Nitric acid	Precipitation	. No effect
Subcarbonate of potash .	No effect	. Precipitation
Water	Precipitation	. No effect.

and carbonic acid; if such combinatio

On comparing these statements it will be evident that the solution procured at Apothecaries' Hall is an oxinitrate of iron, containing much nitric acid in excess; probably also some nitre. I have already stated that the alcali now ordered is more than requisite to saturate the acid, although not sufficient to dissolve the oxide of iron; the medicine which I have just described must therefore have been prepared by increasing the quantity of acid as advised by Dr. Powell, omitting a proportional increase of alcali to saturate it; the solution of iron being then added to that of the alcali until no further effervescence ensued.

Dr. Powell observes that there is "reason to believe" that this preparation "is a triple salt, formed by the union of nitric acid with red oxide of iron, and with potash;" but it appears to me to be merely a solution of peroxide of iron in subcarbonate of potash, perfectly analogous to that of magnesia in this alcaline salt.

The only advantage which the College seem to have had in contemplation when introducing this preparation, was that of exhibiting oxide of iron in solution with an alcali; this intention is however entirely frustrated by the decomposition which occurs upon the addition of water to this solution. If however it be desirable to possess a preparation of iron which may be employed in this manner, it is evident from what I have stated of the properties of solution of tartarized iron, that it can be mixed with subcarbonate of potash without any decomposition; and consequently that it may be conveniently adopted as a substitute for this very uncertain preparation.

I have observed when treating of carbonate of iron that I could not obtain a solid compound of peroxide of iron and carbonic acid; if such combination could exist, it

might be expected to occur when the peroxide is precipitated from its solution in subcarbonate of potash by water, in the manner above described; as however the precipitate so procured is soluble even in its moist state in muriatic acid without effervescence, it is certainly mere peroxide of iron.

## TINCTURA FERRI MURIATIS.

In the Pharmacopæia of 1787, this tincture was directed to be prepared by digesting rust of iron in muriatic acid during three days, and then evaporating the solution to one third of the weight of the acid employed. As the rust of iron is rejected from the present Pharmacopæia, the carbonate is substituted for it, and the evaporation which was not only unnecessary but wasteful and noxious, is now very properly omitted: probably in imitation of the method contained in the Dublin Pharmacopæia.

Half a pound of carbonate of iron is now ordered to be digested during three days in a pint of muriatic acid, and three pints of rectified spirit to be added to the filtered solution. I have shown at p. 10, that the muriatic acid of the present Pharmacopæia, instead of being of sp. gr. 1.17 as directed, is only of 1.142; and that its solvent power is thereby reduced from 20 to about 17; consequently if acid of the latter strength be sufficiently powerful to dissolve the carbonate of iron, that of the former will be too strong, and the solution will have acid in excess: this although perhaps of no consequence, is actually the case when acid of sp. gr. 1.17 and carbonate of iron containing 14.5 per cent. of carbonic acid are employed.

It appears to me preferable to employ digestion for a short time with heat, to longer digestion without it, in order to avoid any uncertainty in the strength of the solution, which might arise from variation in the solubility of peroxide of iron, dependent upon its different degrees of aggregation.

When muriatic acid of the sp.gr. 1.17, and precipitated peroxide of iron obtained by the method I have recommended, which contains only 3 per cent. of carbonic acid, are made use of, the proportions now adopted in the Pharmacopæia answer extremely well, scarcely any oxide remaining undissolved when the acid has been heated. Dr. Powell observes that "this salt appears to be an oxymuriate of iron; the red oxyd of iron employed, becoming on its combination black oxyd, and giving over its superabundant oxygen to the muriatic acid. Sulphuric acid added to it detaches oxymuriatic acid. Heat drives over oxymuriatic acid; and in the latter instance although the red oxyd was used for its preparation the black oxyd remains behind."

Without entering minutely into the consideration of the nature of the oximuriatic compounds, upon which the active genius and elaborate research of Mr. Davy have lately thrown so much unexpected light, I shall content myself with observing, that it would be difficult to collect in the same space more assertions contradicted at once by theory and by fact, than those which I have now quoted. At the time Dr. Powell wrote (scarcely two years since) it was indeed supposed that this preparation was an oximuriate of iron, but not an oximuriate according to the meaning attached to the word by Dr. Powell: it was imagined to be a muriate of red oxide, the term oxi—, prefixed to muriate, referring to the state of the oxide,

and not to that of the acid. Moreover sulphuric acid expels common and not oximuriatic acid, as is readily perceived by the smell, and demonstrated by litmus, the colour of which it reddens instead of destroying it; and when decomposed by heat common muriatic acid is evolved, and unmagnetic oxide of iron remains in the crucible.

## VINUM FERRI.

I have shewn when treating of ferrum tartarizatum that the solubility of iron depends very much upon its softness; and this observation should be kept in view in making this preparation. I have found that a pint of this wine prepared with Sherry and filings of foft iron, contains about 22 grains of peroxide of the metal.

#### HYDRARGYRI OXYMURIAS.

Most of the mercurial preparations are made on the large scale only, and each manufacturer probably has some peculiarity of manipulation, and occasionally varies the proportions of the materials employed; on this account I have not judged it requisite to subject the methods of preparing these medicines to so rigid an examination as those which are usually prepared by chemists and apothecaries.

With respect to the mode of preparing hydrargyri oxymurias, I may remark that it is useless to employ purified mercury, because those impurities which remain after distillation are not likely to rise during sublimation. The quantity of muriate of soda directed to be used for the decomposition of the subsulphate of mercury is much too large: Dr. Powell has stated that three parts of sulphuric acid are capable of decomposing four parts of muriate of soda; yet in this preparation, after a large portion of 30 parts of sulphuric acid has been decomposed by ebullition with 24 parts of mercury, it appears to be supposed that the remainder can decompose 48 parts of muriate of soda; although the 30 parts originally employed could decompose only 40.

## HYDRARGYRI OXYDUM CINEREUM.

This preparation is now introduced into the Pharmacopœia; of all mercurial medicines it is perhaps the most uncertain, and the method of procuring it which the College have adopted is very objectionable. One ounce of calomel is directed to be decomposed by being boiled in a gallon of lime water, consequently a small product is obtained by employing a large bulk of solution, which renders the process inconvenient.

This medicine is subject to great variety of aspect, according to the mode used in preparing it; when lime water is employed the oxide is of a greenish colour, which is derived, as observed by Mr. Davy, from the conversion of a part of the protoxide of mercury into peroxide by the decomposition of the atmospheric air contained in the lime water; and this colour may be imitated by mixing a small quantity of red oxide of mercury with the black oxide. When solution of potash is employed the several inconveniences attendant upon the use of lime water are avoided, and a blackish coloured protoxide is obtained without heating the solution. As potash is much more

coluble than lime, it is scarcely necessary to employ one tenth part of the quantity of water; this not only renders the process more convenient, but the quantity of air contained in the water being less, very little of the oxide, perhaps none of it, is converted into peroxide.

In the Edinburgh and Dublin Pharmacopæias, it is directed to decompose nitrate of mercury by subcarbonate of ammonia, and this method is still more objectionable than that of the London College; for if too much heat be employed in dissolving the mercury, oxinitrate of mercury is produced: this forms a triple compound with ammonia; which being white, renders the precipitate of a lighter colour according to the proportion of the mercury converted into peroxide. Protoxide of mercury is very readily converted into peroxide by the application of heat, it is therefore evident that it ought to be dried by exposure to the air, otherwise its activity may be much increased: I learn from Dr. Powell's table of doses that five times as much of the black oxide of mercury may be exhibited as of the red oxide.

Some practitioners intending to decompose calomel, direct it to be simply washed with a solution of subcarbonate of potash; but in proportion as the alcali contains carbonic acid its action is prevented, and a greyish powder is produced, which consists of calomel mixed with a small quantity of black oxide of mercury. It is evident from what I have stated that the following substances are occasionally employed instead of the black protoxide, viz. a mixture of protoxide and peroxide of mercury; of protoxide, carbonic acid, and nitro-ammoniacal peroxide; or of calomel and protoxide.

## HYDRARGYRUS PRÆCIPITATUS ALBUS.

To make this substance, half a pint of liquor potassæ subcarbonatis is directed to be added to a mixed solution of half a pound of oximuriate of mercury and muriate of ammonia. This quantity of subcarbonate of potash is not quite sufficient to decompose the mercurial salt; and if more than is requisite be employed, ammonia is evolved: this circumstance shows that too much muriate of ammonia is made use of. I find indeed that one tenth part of the quantity directed is sufficient to convert the whole of the oxide of mercury into a yellowish white precipitate; but that to render the product perfectly white it requires about half the quantity employed by the College.

# LIQUOR PLUMBI ACETATIS.

As the superacetate of lead is retained in the Pharmacopœia, the necessity of ordering this solution is extremely questionable. It was probably intended that the method of preparing it should resemble that of the former Pharmacopœia, since Dr. Powell renders "decoque ad libras sex" by "boil to six pints;" an instance of the uncertainty attendant on the alteration of the names of measures, to which I have before adverted.

A pint of distilled vinegar of sp. gr. 1.007 will dissolve about an ounce and a quarter of litharge, consequently a gallon will take up only about ten of the 28 ounces directed to be employed. Yet the waste of litharge is not the only loss occasioned by using too large a quantity of it; for the undissolved portion is so much in-

creased in bulk by the action of the vinegar upon it, that a considerable quantity of the solution remains diffused through it.

Dr. Powell observes that "this is a dense solution of a deep brown colour." Now I have frequently prepared this solution, and have generally observed that it is of a straw colour with a slight admixture of green, and of sp. gr. 1.22. I was consequently struck with the incorrectness of Dr. Powell's description; and as it was evident that he had adopted it upon the authority of others, and not in consequence of his own experiment and observation, I procured this solution from the avowed source of Collegiate pharmaceutical knowledge. Upon examining the solution obtained at Apothecaries' Hall, I did indeed find that it was "a dense solution," its sp. gr. being 1.31 instead of 1.22; and its colour was truly "deep brown." The difference of these preparations being considerable, I procured some distilled vinegar and litharge from Apothecaries' Hall, in order to examine whether any peculiarity which they might possess would explain the dissimilarity. But the difficulty was rather increased than diminished, for the preparation now obtained possessed even rather less colour than that which I had before made; and its sp. gr. very nearly resembled it, being 1.25 instead of 1.22.

Suspecting that the dark colour of the solution above described might be derived from the use of vinegar containing colouring matter, I boiled some litharge in undistilled vinegar; and the solution obtained was certainly of a deeper yellow colour than that made with distilled vinegar, but still not at all verging to brown. I now prepared this solution with the acid which remained in the vessel after the distillation of vinegar, and which

consequently contained a very large quantity of colouring matter: by employing this I succeeded in obtaining a solution, exactly resembling that from Apothecaries' Hall in colour, but by no means equal to it in sp. gr.; instead of being 1.31 it was only 1.21.

Abstractedly considered the colour of this solution is not of the slightest consequence, but as every circumstance which tends to render it difficult to identify a preparation is of importance, the consideration even of colour should not be neglected. In addition to this, when vinegar which contains much extract is employed, it acts slowly upon the oxide of lead dissolved, and by continual precipitation the solution is not only rendered weaker, but of uncertain power. I have bestowed more minute attention upon this preparation than its importance might seem to demand; but I have done so because those errors which are sanctioned by authority are doubly productive of mischief, and their detection should be followed by exposure.

#### PLUMBI SUPERACETAS.

The method proposed for the preparation of superacetate of lead is very extravagant; and its introduction is useless, because that which is made for ordinary purposes at a small expense, may be rendered perfectly fit for medicinal use by solution and crystallization.

#### ZINCI OXYDUM.

This oxide as formerly prepared, frequently contained small portions of metallic zinc, which rendered it unfit for external, and very ineligible for internal use; this inconvenience is now directed to be removed by elutriation, which although a tedious operation, is certainly an improvement; for which the College are probably indebted to the Edinburgh Pharmacopæia.

I have found that oxide, or rather carbonated oxide of zinc, containing about 12 per cent. of carbonic acid, may be advantageously prepared by mixing a hot solution of 8 parts of sulphate of zinc with a solution of 5 parts of subcarbonate of potash; they should be kept hot for a short time, in order to expel the carbonic acid, as I have recommended in the preparation of oxide of iron. By this process a very light white product is obtained. If the sulphate of zinc should accidentally contain any oxide of iron, it may be separated by adding potash to the solution previous to the precipitation by the subcarbonate.

### ZINCI SULPHAS.

In the late Pharmacopæia common white vitriol was directed to be purified and kept under the name of zincum vitriolatum purificatum: the College have now ordered the salt to be prepared; and for that purpose have adopted the process of the Edinburgh Pharmacopæia, without any material alteration. This process answers very well. I may however remark that the sulphuric acid is capable of dissolving more than the assigned portion of zinc.

#### OLEA DISTILLATA.

The oils of anise, camomile, carraway, juniper, origanum, rosemary, and pimento, are usually imported into

this country; instead therefore of giving directions for their distillation, the College should have placed them, as well as the oil of cloves, among the articles of the Materia Medica. It is now directed that the dried herb should be employed in distilling lavender, peppermint, spearmint, and pennyroyal: no advantage would arise from this alteration, and it would be attended with so much inconvenience that it never will be adopted; whilst the dried herbs should be and are almost invariably employed for the preparation of distilled waters, the product being much more grateful than that obtained by using fresh plants. The addition of spirit to distilled waters is altogether useless.

### INFUSUM ROSE.

I have remarked at p. 24 that this is one of those compounds which have undergone considerable and essential alteration, without any accompanying change of nomenclature to indicate whether the practitioner intends to employ the former or the present preparation.

In the late Pharmacopæia three drachms of dilute vitriolic acid were directed to be added to the infusion obtained by pouring  $2\frac{1}{2}$  pints of water upon half an ounce of rose leaves; in the present Pharmacopæia 3 fluidrachms of dilute sulphuric acid are employed with the same quantity of infusion. I have shewn at p. 21, that the strength of one measure of the new dilute acid is to that of the same measure of the former as 148 to 100; and the difference between three drachms of the old preparation and 3 fluidrachms of the new, is an increase of one half: the strength therefore of the present infusum rosæ is to that of the former as 3 to 2.

## EXTRACTUM COLOCYNTHIDIS.

This preparation is now introduced into the Pharmacopæia. It is scarcely possible to boil the colocynth in the assigned quantity of water; and the extract obtained is remarkably spongy, and very soon becomes hard and mouldy.

## EXTRACTUM COLOCYNTHIDIS COMPOSITUM.

The omission of the spirit formerly employed in making this compound extract, has increased the facility and diminished the expense of preparing it; probably without rendering it less efficacious.

Dr. Powell has erroneously asserted that "of the scammony used in the last [preparation], not one particle was dissolved by digestion in the proof spirit;" for I found that half an ounce of it dissolved rather more than a scruple of scammony with the assistance of very slight heat, and seemed capable of dissolving much more.

The formula now given appears to be adopted from the Dublin Pharmacopæia.

## EXTRACTUM HUMULI.

To prepare this extract, which is now introduced into the Pharmacopæia, half a pound of hops is directed to be boiled in a gallon of water to half. This quantity of water is considerably too small.

## EXTRACTUM OPII.

In the Pharmacopæia of 1787 opium was directed to be purified by digestion in proof spirit with a gentle heat; and after a distillation of the spirit from the solution, the opium was to be obtained by evaporating the water. Instead of this method half a pound of opium is now directed to be macerated in three pints of water, and the solution is to be evaporated until the extract has acquired a proper consistence.

Although the former method is not the best that could have been devised, yet it appears to me very preferable to the present; and adverting to the statement which Dr. Powell has made of the different powers for dissolving opium, which spirit and water possess, it seems rather singular that the present method should have been adopted. He states that proof spirit assisted by heat dissolves 9-12ths of opium, and that water, although heated, dissolves only 5-12ths; after having made these observations it must be supposed that Dr. Powell was aware that 4-12ths more of the extractive matter of crude opium were obtained by the method of the late Pharmacopoeia, than by the present process. Now this portion either is narcotic or it is not: if it be narcotic, why have the College directed so large a part of so expensive a drug, and a part too which they have hitherto employed, to be rejected? If on the other hand it be not narcotic, why has Dr. Powell stated that the dose of the new preparation is not greater than that of crude opium, of which a large portion is known to consist of insoluble impurity; and of the extract of which, according to this supposition, 4-12ths must be inert?

Seventy two parts of crude opium which had been

dried by steam until pulverizable, were digested in the manner, and with the proportion of cold water now directed in the Pharmacopæia; the residuum after being dried as the opium had been, weighed 42 parts, which left after being boiled in water, 33 parts; and a portion of this being digested with heat in rectified spirit, left such a residuum as indicated that had the whole been so treated 26 would have remained undissolved. By these experiments it appears that after cold water had dissolved 30 parts, hot water and spirit dissolved 16 parts, proving that by the method of the new Pharmacopæia, 16 of 46 parts of extract obtainable from 72 parts of opium, are lost.

The solution obtained by digesting opium in cold water, and that procured by treating the residuum of the cold infusion with ebullition, differ very materially in their sensible properties; the former is of a deep brownish red colour, remains perfectly transparent for a considerable length of time, and smells strongly of crude opium; the latter solution is of a pale brown colour, deposits on cooling the greater part of what had been dissolved, and has very little smell, and that not at all resembling crude opium. The solution procured by digesting the residuum with heat in rectified spirit, after hot water had dissolved all that was soluble in it, is of the usual colour of tincture of opium; and is decomposed by the addition of water, which occasions an abundant yellowish white precipitate.

Unless it can be proved that the resinous part of the opium ought to be rejected as inert or prejudicial, or that there are impurities in opium which are insoluble in cold water, but readily soluble in hot water and in spirit, it is evident, from the experiments above stated, that the best method of purifying opium is to boil it in water as long.

as any portion of it continues to be dissolved; then to digest the residuum in rectified spirit with heat; and lastly to mix the watery and spirituous extracts, after having evaporated part of the water, and distilled the whole of the spirit.

## ALKOHOL.

There does not appear to have been any sufficient reason for introducing this preparation; it is employed only to dissolve mastich in preparing the spiritus ammoniæ succinatus, and rectified spirit answers equally well. According to Chaussier, rectified spirit consists of about 90 parts of alcohol of sp. gr. .798 and 10 parts of water; and when its sp. gr. is from .830 to .815, as directed in the Pharmacopæia, only about half the quantity of water is abstracted.

If it should in future be thought expedient to procure a stronger spirit than the rectified, it would be better to employ muriate of lime than subcarbonate of potash, for thus alcohol of sp. gr. 806 may be obtained; and probably the sp. gr. may be yet more reduced by the muriate. Yet if the College persist in using subcarbonate of potash, their unnecessarily tedious method of employing it may be readily improved; for by mixing dry but not hot subcarbonate with rectified spirit, and proceeding immediately to distillation, without the digestion ordered by the College, I procured alcohol of sp. gr. 816.

# SPIRITUS AMMONIÆ,

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## SPIRITUS AMMONIÆ AROMATICUS.

In the Pharmacopæia of 1787, spirit of ammonia was prepared by adding three pints of proof spirit to a mixture of four ounces of muriate of ammonia and six of impure subcarbonate of potash; one half of the spirit being distilled constituted the spirit of ammonia; and the compound or aromatic spirit was prepared by adding oils of lemons and cloves to a portion of this product. These processes are both objectionable, for three parts of subcarbonate of potash not being sufficient to decompose two of muriate of ammonia, a part of the latter salt must be wasted; the loss of spirit is however still more considerable. Proof spirit is composed of about 19 parts of rectified spirit and 13 parts of water; three pints consequently contain about 28 fluidounces of the former, of which 24 only were directed to be distilled. The compound spirit was not only coloured but frequently turbid, owing to the usual impurity of the oils of lemons and cloves. The modes of preparing these medicines have now undergone considerable alteration; and the new processes derive from novelty their only merit, and from mischief their sole importance.

The following are the directions for preparing the "Spiritus Ammoniæ:

"Take of rectified spirit, two pints, solution of ammonia, a pint.

" Mix."

The spiritus ammoniæ aromaticus is prepared by adding

a fluidrachm of oil of lemons and of oil of cloves to a pint of the simple spirit.

Dr. Powell asserts that "alkohol dissolves ammonia, but not its carbonate, water dissolves both; and it is further intended that the present spirit shall be of sufficient concentration to dissolve certain volatile oils in a subsequent preparation. The former preparation," he continues, "appeared very uncertain as to strength, and a large portion of carbonate of ammonia sublimed over which was not dissolved; the object of the present change has therefore been to obtain a more definite article than the former."

Now the first of the above quoted allegations is proved to be erroneous by a very simple experiment. I have stated that in the former preparation only 24 of 28 parts of the rectified spirit directed to be used are obtained by distillation; the product must therefore be considered as very highly rectified spirit; and yet if some of it be added to a solution of muriate of lime, a copious precipitation of carbonate of lime ensues.

Neither have I observed that the former preparation was at all uncertain in strength, but on the contrary the results of my experiments coincided to prove a very sufficient degree of uniformity in this respect, as I shall presently more particularly show; whereas the present process has in addition to its other faults, that of uncertain power in an eminent degree. Under the head of liquor ammoniæ I have stated that I found its sp. gr. to be .904, whilst that from Apothecaries' Hall had a sp. gr. of .988, and the power of the first of these preparations is nearly five times as great as that of the latter; consequently the aromatic spirit of ammonia prepared with solutions varying as these, will differ equally in power.

In order to compare the strengths of the new and the former preparation, I again made the liquor ammoniæ; but its sp. gr. instead of being as before .904, was .914, and it was consequently weaker than that which had been previously obtained: with this I made the spiritus ammoniæ aromaticus as now directed, and added a fluidounce of it to a portion of muriatic acid which previous experiment had shown to be capable of decomposing 240 grains of marble; the solution was strongly acid, and I therefore added some pieces of marble to it, having first noted their weight. After heating the solution to ebullition for a considerable time, it appeared on drying the undecomposed marble that 145 grains had been dissolved; consequently the saturating power of a fluidounce of the spiritus ammoniæ aromaticus is equal to that of 95 grains of marble: this experiment was repeated with scarcely any variation.

I now treated the spiritus ammoniæ compositus of the late Pharmacopæia in a similar way, and the mean of two experiments which varied but little, showed that its saturating power was only equal to about 32 of marble, and consequently that the new preparation when made with liquor ammoniæ of sp. gr. .914, which is far from being the strongest which the Collegiate process is capable of yielding, contains almost precisely three times as much ammonia as an equal quantity of the former medicine. Nor is this all: the ammonia in the new preparation not being combined with any carbonic acid, is rendered much more acrid and powerful; and the difference between the doses of the two preparations is consequently still greater than that of their saturating power. Notwithstanding this vast difference, Dr. Powell, has stated the dose of the new preparation to be one fluidrachm; which is equal to

that of the former preparation mentioned by Dr. Latham: but it is still more extraordinary that Dr. Powell should have stated the dose of tinctura guaiaci ammoniata, and of tinctura valerianæ ammoniata, to be two fluidrachms; for unless it be granted that guaiacum and valerian destroy the power of half the ammonia, this quantity, even upon Dr. Powell's own authority, must contain a double dose; and it follows also that in giving two fluidrachms of the present volatile tinctures, six times as much alcali must be exhibited, as in a fluidrachm of the former spiritus ammoniæ compositus, and that the difference in power would be greater even than this. As a drachm of the spiritus ammoniæ compositus of the late Pharmacopæia contained a sufficient quantity of ammonia to be exhibited for one dose, and as I have already shown that it is combined with some carbonic acid, it appears to me that the process may be so modified as to obviate the objections which I have made to it; and I shall now show that the employment of carbonate of ammonia is not attended with the uncertainty which Dr. Powell attributes to it.

On examining the sp. gr. and solvent power of the products of two experiments, conducted as ordered in the Pharmacopæia of 1787, I found the sp. gr. of each was .845, and their saturating powers respectively 33.5 and 31; and I believe it will not be easy to attain to a greater degree of exactness in experiments of this nature.

It is well known that when hot water is poured upon subcarbonate of ammonia, it is partially decomposed, carbonic acid being evolved with considerable effervescence. It appeared to me that advantage might be taken of this circumstance in making the preparation now under consideration. Having ascertained the saturating power of

subcarbonate of ammonia, I conjectured that, making the necessary allowance for the loss unavoidable in the operation, about 12 drachms would yield as much ammonia combined with some carbonic acid, as exists in a pint of the former spiritus ammoniæ aromaticus. I accordingly put nine drachms of powdered subcarbonate of ammonia, 12 fluidounces of rectified spirit, and 3 of water, with the due proportion of the aromatic oils, into a retort; and when 12 fluidounces were distilled, I stopped the process and examined the product. It seemed to resemble the spiritus ammoniæ compositus exactly, being of a very sufficient degree of strength, of an agreeable odour, and perfectly colourless and transparent. Its specific gravity was .854, being only about one part in 100 heavier than the preparation which it was intended to resemble; and its saturating power, instead of being 32, was about 27; consequently at least one-fifth more subcarbonate of ammonia ought to have been employed: this experiment was repeated with slight variation in the result. The product above described answered extremely well for preparing the tinctura guaiaci ammoniata, and the method succeeded perfectly in making the spiritus ammoniæ fœtidus, on putting assafætida into the retort.

On account of the inconvenience and expence of obtaining liquor ammoniæ, the method which I have just proposed is evidently much preferable to that now introduced into the Pharmacopæia for preparing spiritus ammoniæ: and a still stronger recommendation is, that a product may be obtained which in power and properties is similar to that of the late Pharmacopæia, whilst the process is much to be preferred. In the first place the quantity of subcarbonate of ammonia which I should recommend to be employed, is rather less costly than

the muriate of ammonia and subcarbonate of potash used in making equal quantities of the spirit; and as already noticed, one-seventh of the rectified spirit made use of remains in the retort, whereas in the method which I have advised the whole of it is distilled: moreover to obtain one pint and a half of product, 3 pints of fluid and 10 ounces of solid materials were employed; whilst to procure an equal quantity of spirit, I use less than two pints of fluid, and but little more than  $2\frac{1}{2}$  ounces of solid ingredients: consequently smaller and less expensive vessels and less fuel are required.

I shall close my observations upon this preparation with stating that an instance has fallen under my notice, in which strangulation was nearly induced by the exhibition of the new preparation; the physician who prescribed it not being aware of its vast accession of power and of its extreme causticity.

#### TINCTURA MYRRHÆ.

A very injurious alteration has been made in the method of preparing this tincture; the spirit and water, in the proportions now directed, form a mixture which is weaker than proof spirit; and when myrrh is digested in it, a tincture is obtained which by no method whatever can be rendered clear. I cannot even imagine any reason for the change, and the only apology which it will admit is that it was untried.

# TINCTURA OPH.

The method of preparing this tincture has undergone considerable change, and it appears to me that the alter-

ation has been productive of no advantage; more especially as the power of the preparation has been materially altered.

In the Pharmacopæia of 1787, opium purified by solution in proof spirit was directed to be used in making this tincture; in the present Pharmacopæia an equal weight of crude opium has been substituted for the purified opium before employed. I have made several experiments upon each tincture, in order to ascertain, as nearly as circumstances will admit, the quantities of opium contained in similar portions of each.

Some opium taken from one mass was dried by the heat of steam until pulverizable. Three portions of tincture were prepared by digesting five drachms of this opium in eight fluidounces of proof spirit of sp. gr. .930, as directed in the Pharmacopæia. The sp. gr. of the tincture obtained was .952, and the residuum of the portions of opium after having been dried, showed that the opium contained in each quantity of the tincture was 217 grains, 214 grains, and 210 grains, giving a mean of 213.6 grains; equal to about 26.7 grains in each fluidounce of the tincture. That this was nearly the quantity of opium contained in a fluidounce of the tincture was also proved by evaporating certain portions of it to dryness by the heat of a water bath, the mean of three experiments indicating 25.6 grains. Taking the mean of these results, 26.7 and 25.6, it appears that one grain of opium is contained in 18.3 minims of the tincture prepared according to the directions of the new Pharmacopæia. I next prepared three portions of the tincture according to the process of the late Pharmacopocia. Five drachms of the opium purificatum durum dried by steam were digested in eight fluidounces of proof spirit

of the sp. gr. .930. The sp. gr. of each portion of tincture was .958. Of the 300 grains of purified opium used in each experiment, 12 grains in two, and 11 in the third remained undissolved: the mean quantity contained in eight fluidounces of tincture being 288.4 grains, or in one fluidounce, 36 grains; so that 13.3 minims contained one grain of opium.

From these experiments it appears that the strength of the former tincture is to that of the present nearly as 3 to 2; but it must be remembered that during the evaporation of the spirit some of the narcotic power of the opium is lost, and if, as Dr. Powell has asserted, "the power of the present tincture is equal to that of the former," the loss must be nearly equal to half the remaining extract: this however is absolutely incredible; and it must I think be admitted that the former method is much more likely to produce a medicine of unvarying strength, than the present, because opium contains unequal quantities of impurity.

#### SPIRITUS AMMONIÆ FŒTIDUS.

This medicine was directed in the late Pharmacopæia to be prepared by distilling  $2\frac{1}{2}$  pints of product from 3 pints of proof spirit, 2 ounces of assafætida, 6 ounces of muriate of ammonia, and 9 of impure subcarbonate of potash. The quantities of muriate of ammonia and subcarbonate of potash are less than those used in distilling an equal quantity of spiritus ammoniæ, in the proportion of about 6 to 6.6; whereas the product to be obtained from an equal measure of proof spirit is greater in the proportion of 5 to 3. The difference between the saturating power of this preparation and the spiritus ammoniæ compositus, I have not thought it

requisite to ascertain; but it is probable that although less of the muriate is used, there is as much ammonia in the fetid as in the compound spirit; for as the former contains much more water, it is of course capable of dissolving a considerable portion of the subcarbonate of ammonia which remains undissolved in preparing the compound spirit.

In the present Pharmacopæia this process is materially altered, and without having tried it I do not hesitate to pronounce that the change is for the worse, because it requires the employment of the liquor ammoniæ. I have stated when treating of spiritus ammoniæ compositus, that the medicine now under consideration may be prepared by adding assafætida to the ingredients which I have recommended to be used in making the spiritus ammoniæ: and the strength of the product may be made equal to that of the former Pharmacopæia, as I have mentioned with respect to the spiritus ammoniæ aromaticus.

### SPIRITUS AMMONIÆ SUCCINATUS.

This preparation must surely be useless: in Dr. Powell's table it is stated, that 30 minims of oil of amber are a dose, and in this preparation, in order to give one minim of the oil,  $2\frac{1}{2}$  fluidounces of liquor ammoniæ must be exhibited: with a dose of oil of amber, therefore, 1800 of Dr. Powell's doses of liquor ammoniæ are given; a quantity which, reduced to the strength of the old preparation, as made at Apothecaries' Hall, was considered by the College in 1787 as sufficient for twenty-five doses a day for a twelvemonth; and Dr. Powell's dose of this preparation contains scarcely 1-13coth part of the quantity he assigns for the dose of oil of amber.

#### ETHEREA.

The Pharmacopæia of 1787 directed the spiritus ætheris vitriolici to be prepared by mixing equal weights of sulphuric acid and rectified spirit, and submitting the mixture to distillation until sulphurous vapours began to arise. The æther vitriolicus was made by adding a fluidounce of aqua kali puri to two pounds of the ætherial spirit obtained by the process above described, and fourteen fluidounces of this mixture were directed to be distilled and preserved.

These processes are unnecessarily expensive; for although the distillation is directed to be continued for obtaining oil of wine after the spiritus ætheris vitriolici is procured, yet the residual acid is sufficiently strong to ætherize an additional quantity of spirit; and the College have now very properly adopted the method of the other Pharmacopæias in directing a second portion of spirit to be poured upon the acid remaining in the retort. The method of obtaining what was denominated in the late Pharmacopæia æther vitriolicus, is still more wasteful; the sp. gr. of the first product being, as I shall hereafter show, about .768, two pounds of it would be equal to about 34 fluidounces; yet of this quantity, 14 only, as already stated, were to be distilled for use.

In the present Pharmacopæia equal weights of sulphuric acid and spirit are to be employed, as in the former process; but the distillation is now to be continued "until a heavier fluid also begin to pass over, and appear under the æther in the bottom of the receiver;" but as it is also stated that the receiver is to be "kept cold by immersion in ice or water," it would be difficult for

difference between two fluids varying only in gravity. The production of this heavy fluid cannot moreover be admitted as a criterion that the process ought to be discontinued, because it does not invariably occur in similar circumstances. Of two experiments conducted at the same time, and in the same manner, one product consisted of two fluids, and the other was one homogeneous fluid; consequently the new method is altogether uncertain. I apprehend it may be safely concluded that the authors of the present, as well as of the late Pharmacopæia, intended that the distillation should be continued as long as the product remained tolerably free from sulphurous impregnation.

I have already mentioned that the College have now, in imitation of the Edinburgh and Dublin Pharmacopæias, ordered a quantity of spirit to be added to the acid residuum of the first operation; but with strange, though not unparalleled inconsistency, the second portion is directed to be measured instead of being weighed, as is the case with the first; and this is the more extraordinary, because the quantity to be made use of very little exceeds half the weight of that originally employed; and in the Dublin Pharmacopæia precisely half the first quantity is directed to be used. It may seem unnecessarily operose to use a certain portion of spirit with a quantity of acid in two distillations instead of one, but the fact is that a portion of alcohol always passes over unchanged, and this is greater when the concentration of the materials is diminished. I found that the first four fluidounces yielded by distilling 16 ounces of sulphuric acid and an equal weight of spirit, were of sp. gr. .779; whereas the second portion of four fluidounces was of sp. gr. .753.

To one pound of sulphuric acid an equal weight of rectified spirit was added, and by submitting this mixture to distillation, I procured fl. oz. 8 of product, consisting of fl. oz.  $7\frac{1}{2}$  of æthereal spirit, of sp. gr. .768, and half a fluidounce of heavy fluid; after the addition of fl. oz. 8, the second portion of spirit, as directed, I procured fl. oz. 8 of æthereal spirit, of sp. gr. .807, and as much heavy fluid as at first.

The College do not seem to have been aware that the products of the first and second distillation differed in sp. gr., for they have not directed them to be mixed as ought to have been the case; but it would follow from the mixture of the two products above described that the sp. gr. of the present æther sulphuricus is about .788, whereas the former spiritus ætheris vitriolici, which must be considered as analogous to the first portion obtained, had a specific gravity of .768.

I repeated this experiment, employing 16 oz. each of acid and spirit; the sp. gr. of the former was only 1.837, instead of 1.850, as directed in the Pharmacopæia, and the sp. gr. of the latter, instead of being .835, was .830. Twelve fluidounces of product were obtained which consisted of

4 fl. o	z. of	æthere	al spin	it, of	sp. gr.	.779
4 .					Letie	.753
21/2 ·	. of	yellow	sulphi	urous	do. do.	.784
I 1 .	. of	heavy	fluid			.981

On again repeating this experiment I found the sp.gr. of the first four fluidounces was .773, and that of the second .751, which very nearly resemble those above given, but a portion of the contents of the retort having accidentally passed over into the receiver, I was prevented from examining the after products, and I employed the acid residuum of both experiments in attempting to procure the oleum æthereum. By mixing the three first portions of the experiment described in the preceding page, 10½ fluidounces of æthereal spirit of the sp. gr. .770 will be obtained, which is a product nearly the same in quantity and in specific gravity as the result of the first operation described. It must however be stated that this coincidence is by no means to be expected, or attained to on every occasion.

The Dublin College direct that fl. oz. 20 should be distilled from a mixture of 32 ounces of sulphuric acid with an equal weight of rectified spirit; and from the experiments which have already been stated, as well as from those I am about to relate, it will appear that this process may be very conveniently adopted; especially in connection with a method of rectification which I shall presently describe and recommend. The directions of the Dublin College are imperfect in not giving any rule for the continuance of the second distillation, and this I shall endeavour to supply. Sixteen oz. of sulphuric acid of sp. gr. 1.837, mixed with a similar weight of rectified spirit of sp. gr. .830, were quickly made to boil; and in about 3 hours I obtained fl. oz. 10 of product, of sp. gr. .791, unaccompanied by any heavy fluid: eight ounces of spirit being added to the acid in the retort, I procured in about 21 hours, fl. oz. 9 of sulphurous æthereal spirit, of sp. gr. .872, likewise without any heavy fluid.

On repeating this experiment, I procured by the first distillation fl. oz. 10 of æthereal spirit without any heavy fluid, its sp. gr. was very nearly the same as that of the former experiment, being .794, instead of .791: after the second portion of spirit was added, I obtained fl. oz. 8½

of sulphurous æthereal spirit, the sp. gr. of which was .846; that of the former experiment being .872: the time occupied in the distillation was nearly the same in these, as in the former experiments.

The College have directed that the acid and spirit should be made to boil as quickly as possible: I am apprehensive that no advantage and probably some detriment arises from speedy ebullition; for when quantities of materials similar to those which I have described as yielding fl. oz. 10 of product, of sp. gr. .791, in about three hours, were allowed to be nearly three times as long in giving an equal quantity, I found that its sp. gr. was .782.

The æthereal spirit obtained in the above described processes is a mixture of very different fluids, and the object of the rectification directed is to separate the æther. It is well known that æther is soluble in water, and it may be safely concluded that water is soluble in æther; as is indeed actually the case: its presence is therefore to be more especially suspected in those products which consist of two different fluids; for the heavier appears to be merely water impregnated with æther and sulphurous acid, and the supernatant fluid must consequently be considered as having taken up as much water as it is capable of combining with: it is evident also that sulphurous acid and alcohol constitute a part of the product. To render the æther pure therefore, it must be separated from alcohol, sulphurous acid, and water: several processes have been recommended for this purpose, and among others the use of manganese; but this substance is less powerful than potash in combining with sulphurous acid, and it has no power to combine with water or with alcohol. The London College direct the æther rectificatus to be prepared by dissolving one ounce of potash in two

fluidounces of water, and adding fl. oz. 14 of the æthereal spirit to this solution: from this mixture fl. oz. 12 of æther are to be distilled. The loss of æthereal spirit, amounting to one seventh, is greater than needful: this method moreover cannot be very efficient in removing water and alcohol; and the æther obtained by it, as procured from Apothecaries' Hall, is of sp. gr. .778.

It appeared to me that it would be an improvement to add solid potash to the æthereal spirit; for having a considerable attraction for water and alcohol, and being totally insoluble in æther, it seemed probable that the potash would dissolve in the former fluids and sink: this supposition I proved to be well founded. To æthereal spirit of sp. gr. .814, I added solid potash until no more appeared to be dissolved; and having separated the æthereal fluid from the solution of alcali, I submitted it to distillation, and continued the operation until only about one tenth of the quantity made use of remained in the retort. The product was extremely fragrant, and was of sp. gr. .750; and indeed I found that by using potash in this way, æther might be obtained of sp. gr. .729.

In the Pharmacopæia of 1787, the first product of what is now called æther sulphuricus, the sp. gr. of which as already mentioned is about .768, was called spiritus ætheris vitriolici: the spiritus ætheris sulphurici is now directed to be prepared by adding, in imitation of the Edinburgh College, two measures of rectified spirit to one of sulphuric æther; so that the present preparation is composed of one part of the former and two parts of alcohol; its power is consequently much diminished, yet Dr. Powell has stated its dose to be only half that of the æther rectificatus.

With respect to the nature of oil of wine, now called

oleum æthereum, the opinions of chemists have been divided; some alleging that it is really a peculiar volatile oil, while others imagine it to be merely æther impregnated with sulphurous acid. After repeated trials I am inclined to adopt the latter opinion, for I have never procured any æthereal product having the appearance of oil, which was not instantly separable into these two constituents by solution of potash.

In the late Pharmacopæia two methods of obtaining this substance were directed; equal measures of alcohol and sulphuric acid being mixed and submitted to distillation, the oily part of the product was to be separated from the sulphurous, and solution of potash' being added to the former, the æther which it contained was to be distilled from it, the oil remaining in the retort. This method I have not tried: the other consisted of adding a receiver to the retort after the distillation of the spiritus ætheris vitriolici; and as that was ordered to be obtained as long as no sulphurous acid was generated, it is evident that the after product must contain some. The product obtained in this way is similar to that which I have described at p. 136, as yellow sulphurous æthereal spirit of sp. gr. .784; and if it be added to a solution of potash, its colour and sulphurous smell are immediately lost, and the æther swims on the surface of the alcaline solution.

In the present Pharmacopæia the following are the directions for preparing æthereal oil, and it may be considered as resembling in every material respect the first of the above described processes: "After the distillation of sulphuric æther, carry on the distillation with a less degree of heat, until a black froth begins to rise, then immediately remove the retort from the fire. Add suffi-

cient water to the liquor in the retort, that the oily part may float upon the surface. Separate this, and add to it as much lime water as may be necessary to neutralize the adherent acid, and shake them together. Lastly, collect the æthereal oil which separates."

I have repeatedly attempted this process, and have uniformly failed in obtaining æthereal oil or any æthereal product whatever: no reasonable preference can be assigned for the use of lime water in place of potash. It is indeed true as Dr. Powell states, that potash unites with oil, and so also does lime water; but if a sufficient quantity only be used to saturate the acid, there is no danger of its combining with the oil. The quantity of lime water which this process requires is enormous; I found that a fluidrachm and a half from the surface of the liquid obtained as above directed, was sufficient to saturate two pints of lime water, consequently had nearly the whole, instead of no part of it, been oil of wine, it would have required ten pints of lime water to procure a fluidounce of oil. Having frequently observed that the yellowest and most sulphurous portions of æther, which are last distilled, are heavier than the earlier portions, it appeared to me that it might be possible to produce this fluid immediately, by accomplishing the decomposition of the sulphuric acid more quickly than it is usually effected by the alcohol. With this view I added some sugar to spirit and a large proportion of sulphuric acid; sulphurous acid was consequently soon formed, and I procured yellow sulphurous æther, of an oily appearance, part of which sunk in the fluid distilled along with it; but by the use of solution of potash I found that it consisted of æther combined with sulphurous acid. Notwithstanding all these experiments, I do not mean positively to deny the existence of oil of

wine; but every product which I have prepared or procured from others, uniformly consisted of the yellow sulphurous æther above described, or of æther coloured by carbonaceous matter, having a slight empyreumatic smell, but not at all sulphurous, with a sp. gr. of about .768; and 32 measures of this yielded by simple distillation 27 of æther, of sp. gr. .747, and sulphuric acid added to the coloured residuum in the retort was immediately decomposed.

So difficult then and uncertain is the production of oil of wine, and so insignificant its employment by the College, that it does not appear to me to be a preparation worth retaining. I find by Dr. Latham's table that he considered four drops of it a full dose; but as it is now directed that two fluidrachms are to be added to a pint of spiritus ætheris sulphurici, and Dr. Powell limits the dose of this compound to a fluidrachm, it is evident that not one minim is exhibited.

Upon considering the statements now made it appears to me, that it would be advantageous to reject the oleum æthereum, and the following preparations also, æther sulphuricus, spiritus ætheris sulphurici, and spiritus ætheris sulphurici compositus, retaining only, under the appellation of æther sulphuricus, what is now termed æther rectificatus. Some of the reasons for this recommendation will be found scattered through the above remarks; I may however further observe with respect to the present æther sulphuricus, that no advantage can be supposed to attach to it that does not belong more certainly, and in a higher degree, to the æther rectificatus; and that the spiritus ætheris sulphurici cannot be imagined to derive any specific advantage from the alcohol of which it partly consists: it is certainly more soluble than æther in water,

but the latter is sufficiently so to be exhibited in small doses, and large quantities of the former preparation cannot be given without administering twice its bulk of alcohol. For procuring æther I recommend the following process: Mix with 16 oz. of sulphuric acid an equal weight of rectified spirit, and distil about 10 fluidounces; add 8 ounces of spirit to the residuum in the retort, and distill about 9 fluidounces, or continue the operation until the contents of the retort begin to rise, or the product becomes considerably sulphurous. Mix the two products, and if the mixture consist of a light and heavy fluid, separate them: add potash to the lighter as long as it appears to be dissolved; separate the æther from the solution of potash, and distil about nine tenths of it, to be preserved as æther sulphuricus; the specific gravity of which ought to be at most .750.

From the powerful action of the potash already mentioned, it is of but little consequence, except in an economical point of view, what portions of spirit, water, and sulphurous acid are mixed with the æther, since they all unite with the potash, and form a distinct fluid: it is evident that this alcaline solution should be preserved, not only because it contains potash, but on account of the alcohol which it has separated from the æther. The heavy fluid which, as I have mentioned, is a mixture of water and æther, should be preserved, and when a considerable quantity is collected, the æther should be separated by distillation: the residuum left in the retort, even after the second distillation of æthereal spirit, still contains a useful quantity of sulphuric acid.

quantity of water is required to dissolve only two nounds

#### SPIRITUS ÆTHERIS NITRICI.

Half the nitric acid formerly used in this preparation is now advantageously omitted; but fl. oz. 26, the quantity ordered to be distilled from two pints of spirit and three ounces of acid, is too much; when 24 fl. ounces only are obtained, the preparation is scarcely at all acid; it is also colourless, and is of sp. gr. .834, but the succeeding portions are highly coloured and very acid. I learn from Dr. Powell that the proportions now directed are nearly similar to those employed by Hoffman.

## the solution of potash, and distill about nine tended of it, to be preserved as other supplementary the specific gravity

to be discoved deparate the a

In the late Pharmacopæia 29 ounces of sugar were directed to be dissolved in a pint of any fluid from which syrup was to be prepared; in the present Pharmacopæia 30 ounces are ordered to be employed with the same proportion of fluid; an increase which according to Dr. Powell's calculation amounts to one-fifth.

## of The works and SYRUPUS AURANTII.

use it contains potash, but on account of the

Dr. Powell observes that "acid juices dissolve somewhat less sugar than water does;" whilst on the other hand he appears to think that aromatic infusions dissolve somewhat more; for a pint of infusion of orange peel is directed to dissolve 3 pounds of sugar, whereas an equal quantity of water is required to dissolve only two pounds and a half.

#### SYRUPUS PAPAVERIS.

I suppose it will be allowed that in all the cases in which the capsules of poppies are used, their virtue should be equally extracted; yet it is curious to observe how various are the directions of the College for producing this effect. In preparing the decoctum papaveris, they are directed to be simply bruised and boiled, whilst in making the extract they are not only to be bruised, but to have boiling water poured upon them, and after macerating for 24 hours, the water is to be boiled to half. In preparing the syrup, they are as before directed to be bruised, and to have the seeds taken out, and hot water, as in the last instance, is to be poured upon them, but the maceration is to be for 12 hours only, and the boiling is now to be conducted in a water-bath: if this precaution be needful in preparing the syrup, it can be no less so in making the extract; but the fact is, that it is not requisite in either.

#### SYRUPUS SENNÆ.

The formula for making this medicine very much resembles that of the Dublin Pharmacopæia, in which it must have been inserted without previous experiment, and from which it has been copied without examination.

One pound each of manna and sugar are directed to be dissolved in a pint of infusion of senna and fennel seeds; but it happens very unfortunately that the syrup is extremely thick; that it not only does not flow from an inverted vessel, but that it is also unmoved by repeated percussion. This circumstance appears to be owing to

the crystallization of the manna, for on close inspection crystals of this substance may be observed.

#### CONFECTIO AROMATICA.

It would be much better to employ prepared chalk than oyster shells in making this medicine; indeed the former carbonate may be advantageously exchanged for the latter in every instance, since owing to the hardness of the shells, it is very difficult to reduce them to so fine a powder as that which chalk easily affords.

#### CONFECTIO SENNÆ.

It does not appear that the extractive matter yielded by two ounces of a compound of the stalks of senna and of the husks of coriander seeds, is likely to increase the activity of about 80 ounces of this confection so materially, that it is worth while to boil them in the decoction of liquorice root.

#### UNGUENTUM HYDRARGYRI NITRATIS.

The quantity of nitric acid used to dissolve the mercury, has been very unnecessarily increased from two ounces to two fluidounces. This occasions violent action, and the evolution of much noxious vapour, when the solution of mercury is melted with the lard; and renders the ointment extremely corrosive.

#### LINIMENTUM CAMPHORE,

ET

#### LINIMENTUM CAMPHORÆ COMPOSITUM.

The former of these preparations, consisting of a solution of camphor in olive oil, is now introduced. The name affixed to it will probably occasion some confusion, because in the first editions of the late Pharmacopæia the latter preparation was called simply linimentum camphoræ, and it is yet frequently so denominated.

The method now adopted for making the linimentum camphoræ compositum is objectionable, because it necessarily includes the use of the liquor ammoniæ. It would I apprehend be better made by distilling the spiritus lavandulæ from the same proportion of subcarbonate of ammonia, and in the manner in which I have stated that the spiritus ammoniæ may be most advantageously prepared.

I shall conclude these observations with remarking, that the posological table annexed by Dr. Powell to his translation is very imperfect; and I have occasionally adverted to instances of incorrectness requiring careful revision. The Table also expressing "the relative value in avoirdupois weight of various weights troy," is one unvaried mass of error: it seems to have been intended for the use of those persons who prepare medicines in large quantities, and it has probably been productive of many important mistakes. Three fundamental errors

have been the occasion of this collection of mis-statements; first, the avoirdupois drachm is reckoned to contain 27.975 grains instead of 27.34375; secondly, the avoirdupois ounce is estimated to consist of 8 drachms, instead of 16; and lastly, the troy ounce is stated to be equal to one ounce and 50 grains avoirdupois, i.e. to  $487\frac{1}{2}$ , instead of 480 grains.

FINIS.

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## POSTSCRIPT.

SOME errors have been discovered in the foregoing observations, which appear of sufficient importance to be pointed out and corrected.

The Critical Reviewer (July, 1812), observes that in my remarks upon the experiments detailed in page 67 of Dr. Powell's Translation, I demonstrate and yet deny that the new method of preparing nitric acid is more economical than the former. Now admitting these experiments to be correct, which they obviously are not, I should only have stated that the new product is not of so much greater value than the old as it is represented to be; and that the process for obtaining it is less economical than it ought to have been, if the experiments in consequence of which it is said to be adopted, had really served this purpose.

It has been observed to me that the density of nitric acid is increased by the absorption of nitric oxide. I have proved the truth of this remark by direct trial; and it is possible, as announced in the statement above alluded to, that red nitric acid, of sp. gr. 1.53, may possess less solvent power than pale acid of sp. gr. 150: the opposite opinion therefore which I have advanced, is erroneous.

In page 94 I have committed some errors by copying Dr. Thomson's calculation that 100 parts of peroxide of iron contain 66 of protoxide; the fallacy of which he has admitted in a late number of Nicholson's Journal. According to Sir H. Davy, 143.5 of peroxide contain 129 of protoxide; 28 will therefore give 25 instead of 18.6, as the protoxide contained in 100 of sulphate of iron, and 100 parts of carbonate of iron ought to contain 16.6 of carbonic acid instead of 21 as I have mentioned.

Page 100. In preparing the solution of tartarized iron, I have found it convenient to saturate the last portions of tartaric acid with ammonia; any excess of the alcali being readily dissipated by heat, without occasioning decomposition. The ferruginous taste is by this method rendered scarcely perceptible.

Page 121. I find that Extract of Colocynth may be prepared so as not to become hard and mouldy by keeping, as I have stated, but repeated complaints of severe griping occasioned by the Compound Extract of Colocynth prepared according to the new formula, have not only changed my opinion of its efficacy, but also obliged me to recur to the former more troublesome and expensive method of preparing this medicine.

Page 33, line 19, for it is only .9885, read it is .9855.

Page 124, line 13, for is from, read is raised from.