COMMUNICATION FROM THE SOUTH LONDON PUBLIC LABORATORY—
SIMPLE SUGGESTIONS FOR THE PURCHASE AND ASSAY OF COMMERCIAL CARBOLIC COMPOUNDS USED FOR SANITARY PURPOSES.

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OWING to the extensive use of these compounds by the various local sanitary authorities throughout the country, and the occasional demands on the official analyst for their valuation, it is very desirable, both in the interests of the producers and the consumers, that there should be some simple and definite methods agreed upon for their analysis. Up till the present time we venture to think that if the same sample were sent to any dozen analysts, taken by chance all over Great Britain, not more than two or three would come within measurable distance of each other in the percentage of carbolic acid found, while it is more than probable that the manufacturer's chemist would disagree with all. This state of things is caused:—

(1) By the fact that, very probably, nearly all the operators would employ a process of their own devising, and that, although most would work upon similar lines, yet the details would be so different as to cause a very wide divergence in the finished results.

(2) By the fact that, in commercial mixtures of carbolic acid and the higher phenols, the commonly adopted methods come entirely to grief.

The process of Koppeschaar (although we continually meet with some one who has modified it so as to render it perfect) is totally unreliable for commercial use, because it is full of pit-falls. For example, to begin with, we have the necessity of working on such small quantities that any experimental error is multiplied by something like 1,000; and then, as we are really to a great extent precipitating cresol and calculating it as phenol, we cannot on the face of such a fact expect much truth in the result. This is not, however, all, because the process is not to be depended upon even when dealing with pure medicinal acid. One may set to work to make a series of test experiments, and may theorise that, if it can only be arranged so that we work with the same excess of bromine and for the same time, we are bound to get concordant results. If any one has succeeded in really carrying such reasonable theory into practice without meeting with some unaccountable differences in the very first long series of experiments, then he has been much more lucky than we have ever been, and, so far as we know, we have tried every published modification, besides those resulting from our own experiments.

Another thing which tends to lead to much dissatisfaction in such matters is the tendency of local authorities to contract wildly for powders or liquids "containing (say) 15 per cent. carbolic acid," without getting their medical officer to ask the analyst whether such an article really exists in commerce. The manufacturer takes such a contract with a light heart, because he knows that he can practically put in an acid as tarry and weak as he likes, and argue (if challenged) that he really put in 15 per cent. of "commercial" carbolic acid, and bring reliable witnesses, if needful, to prove they saw it done. Meantime the powder is complained of and sent to the analyst, who is asked to state the percentage of carbolic acid. He does this according to his lights, and naturally takes the words of the contract as literal, and reports the amount of *real phenol* he finds. This being generally microscopic, the authority becomes indignant, and

the manufacturer, in defending himself as above shown, hints that the analyst is incompetent, and generally gets one or two persons on the local board to believe him. If, in such matters, the boards could be induced to consult their analyst before issuing the form of tender, they would then learn that words like those we have above commented upon are absolutely useless in such cases, and that it is ridiculous to ask for a thing which never could be supplied in practice within any reasonable limit of cost.

It may be now taken as a tolerably well admitted fact that, setting aside the necessities of surgery and medicine, the cresol and other higher phenols are just as good for ordinary antiseptic purposes as real carbolic acid itself, and that unless the presence of such congeners be permitted, the cost of disinfectants of this class would be quite prohibitory. It is therefore sufficient that the analyst should see that his board is being supplied with an article which is all made up of mixed phenols, and not with tar oils, containing only a small proportion of such bodies. To ensure this the following, or some similar, form of tender should be employed: "Commercial liquid carbolic acid at — per gallon. This article is not to be darker than pale sherry, and it is to contain not less than 95 per cent. of carbolic and other allied acids obtained from tar, the said acids being in their liquefied form, but free from any excess of water. The said article to be subject to the approval of the Public Analyst (whose decision shall be final) on a sample drawn from each delivery by the Medical Officer. Such analysis is to be carried out and the result communicated to the contractor within six days after delivery of each consignment, and should the result be unfavourable the contractor shall remove the rejected article at his own charge."

Turning now to carbolic powders, we may take it as also a fairly admitted proposition that a commercial acid, such as has been above described, may justly be used in their manufacture, provided that the base of the powder is some siliceous matter, or gypsum, or native limestone, with none of which phenols will combine; but when such acid is employed the use of slaked or caustic lime should be rigorously tabooed. It has been proved that the antiseptic powers of cresol (for example) are entirely masked when combined with an alkali or alkaline earth, and what is true about this body is probably also correct about the other higher phenols present. Analysts looking after the interests of their district should advise a specification worded something like the following:—"Carbolic powder, containing not less than 15 per cent. of the commercial carbolic acid as above described, and having for its base any siliceous or other inert mineral matter with which the acid will not combine. The use of quick or slaked lime as bases are specially to be avoided, and each consignment is to be subject to the same conditions of delivery and approval as in the case of the acid above described."

In some cases a small supply of really pure liquefied carbolic acid is required, and then the simplest contract is for an article which shall correspond in every respect to *acidum carbolicum liquefactum* of the British Pharmacopœia.

Such specifications are now in force in several of the districts served by this laboratory, and since their adoption, together with the processes about to be described, there have been no disputes; moreover, on the few occasions when a parcel had to be rejected, the manufacturer, on making inquiries, found that his own people had been in error, and at once admitted the mistake.

There is nothing more involved in the processes than the following well-known data :—

(1) Phenol, cresol, and their homologues are completely soluble when shaken up with a 5 per cent. solution of sodium hydrate.

(2) Liquefied phenol and the corresponding cresol are insoluble in a saturated solution of sodium chloride.

(3) In the presence of a sufficient excess of alkali, even a largely diluted solution may be boiled down without the slightest appreciable loss of phenol or cresol.

(4) Tar oils and naphthaline are only very slightly dissolved by the alkali, and may be perfectly removed from the solution by agitating it with benzol.

Taking these ascertained facts, we apply them in the following manner, taking each case in turn :—

(1) PROCESS FOR THE VALUATION OF A CARBOLIC POWDER IN WHICH THE BASE IS NOT LIME, AND THE PHENOLS ARE CONSEQUENTLY NOT IN COMBINATION.

Weigh out 100 grammes of the powder, and transfer the same to a flask, and add 400 c.c. of methylated spirit ; then, having introduced a well-fitting cork, agitate for a minute or two at intervals during an hour, and finally set the whole aside to settle. When subsidence is complete, pour or filter off 300 c.c. of the supernatant liquid, which thus represents 75 grammes of the original powder actually taken for analysis. Here it may be noted that in laboratories fitted with the specially large "Soxhlet" apparatus required, a more rapid and economical method is to mix the original powder with bran, and to extract it in such an apparatus, with just sufficient spirit to do the necessary work.

To the spirituous extract of the powder, obtained as above described, 200 c.c. of 5 per cent. solution of sodium hydrate are now added, and the mixed liquids are evaporated to about half their bulk. At this point any tar-oils or naphthaline present will separate out, and are to be removed by filtration. The filtrate, freed from these impurities, is now evaporated down to a bulk not exceeding 50 c.c., and transferred to a specially constructed measuring tube surrounded by cold water, the basin being rinsed out with water, so that the entire amount of fluid in the tube shall be exactly 65 c.c. The tube employed is of peculiar form, capable of holding over 110 c.c., and is specially graduated and stoppered. It is wide at the base, and is narrowed at 65 c.c. from the bottom to such a diameter as to show, when graduated, $\cdot 25$ of a c.c. The first graduation is at 20 c.c. from the bottom, the second at 65 c.c., and then the delicate graduations (in $\cdot 25$ of a c.c.) commence and continue for 45 c.c. more up to 110 c.c. The tube is furnished with a long stirring rod made as thin as possible, and projecting above the tube to a convenient extent. [These tubes are to be procured from Mr. Cetti of Brooke Street, Holborn, who knows them as "Muter's carbolimeters," and the entire apparatus costs 8s. 6d.] Before proceeding to use the tube for the first time, it is necessary to find the allowance to be made for the rod. This is done by filling the tube with brine up to the 75 c.c. mark and then pouring in liquefied carbolic acid up to the 85 c.c. mark. The rod is now introduced and the amount of displacement it causes noted, so that an equivalent allowance may be made on all future experiments.

The measuring tube having been charged, as already described, we now proceed to

add very cautiously, and with constant stirring 25 c.c. of strong hydrochloric acid, and when that is all in, we follow it with a teaspoonful of common salt. All the phenols now rise to the surface, and when the whole is at the temperature of 60° Fah., the volume is read off, which gives the amount of commercial carbolic acid present in the 75 grammes of powder. If a perfectly accurate result be required, it is necessary to remove some of the floating acid with a pipette, take its specific gravity, and correct the volume reading to weight.

(2) MODIFICATION OF THE ABOVE PROCESS FOR USE WITH POWDERS ON A LIME BASE, WHERE THE ACIDS ARE COMBINED.

Before proceeding with the spirit extraction, the weighed portion of the powder is to be treated in a capacious mortar with successive small quantities of diluted sulphuric acid (one in three) until the whole mass has a faintly acid reaction, and then the process applied as above described. It is necessary to be very particular about the treatment in the mortar, because, if the least particle of the powder escapes the action of the acid, the results are vitiated, while at the same time, any marked excess of acid should be avoided. Both pestle and spatula must be thoroughly used.

(3) EXAMINATION OF A SAMPLE OF COMMERCIAL CARBOLIC ACID.

Case 1. The sample is dark in colour.—Put some of the sample into the "carbolic-meter" up to the 20 c.c. mark, then gently add 5 per cent. solution of sodium hydrate up to the 100 c.c. mark, and lastly add 10 c.c. of benzol. Put in the stopper, and having inverted the tube once or twice, plunge it into cold water. Repeat this shaking and cooling until the separation is complete. Read off the volume of the dark layer, which will now have formed beneath the 100 c.c. mark, and the amount of this will give the tar-oils, etc., present in the sample. Provided there is no excess of water, this amount is deducted from 20 c.c., and the difference multiplied by five gives the percentage strength of the sample. Excess of water (which is of very rare occurrence) is best ascertained by adding some of the sample to three times its volume of benzol, when it should dissolve quite clear if there be no such excess.

Case 2. The sample is not darker than pale sherry.—Try if it dissolves nearly clear in four times its volume of 5 per cent. solution of sodium hydrate, and if so, it may be taken as practically free from tar-oils. If not, treat it as above, using however, only a very small fixed volume of the benzol. This acid is apt to contain excess of water, which must be estimated by shaking 20 c.c. of the sample in the "carbolic-meter" with 80 c.c. of saturated solution of sodium chloride, and observing the diminution in volume that will take place if such excess be present.

A fair idea as to whether the acid is chiefly phenol or cresol may always be obtained by applying the bromine reaction, and observing the nature of the precipitate.

(4) EXAMINATION OF A SAMPLE OF PURE CARBOLIC ACID IN THE LIQUEFIED FORM.

In examining samples of this acid, specific gravity practically goes for nothing. The points to determine are:—(1) That it should entirely dissolve to a perfectly clear solution in four times its volume of 5 per cent. solution of sodium hydrate; (2) That, when shaken with an excess of saturated solution of sodium chloride, as already described, it suffers no diminution in volume; (3) When treated with bromine water in excess it gives a fine curdy precipitate, not at all inclined to stick to the tube.

In conclusion, we can only repeat our statement that, if those persons engaged in the examination, and in the general supervision of carbolic antiseptics supplied to public authorities, would only agree to universally adopt some such simple methods and specifications as we have described, there would be universal satisfaction, instead of the disputes now so common, and, in addition to all that, the public money would be spent to infinitely better purpose than it now is in too many districts. We could name at least one district in the Metropolis itself where the present fever epidemic is being fought with so-called "carbolic acid," which is practically all inert tar-oils, and does not contain even 2 or 3 per cent. of the higher phenols, much less any real carbolic acid. It is time that local authorities consulted the public analysts more frequently before entering into such a purely chemical matter as a contract for carbolic articles. It is not to be expected that medical officers can keep such things in order, unless where (as in certain of our own districts) the knowledge of the chemical officer is called into play to check the contractors, and to advise as to forms of contract that can really be carried into practice with full justice to all parties concerned.