

THE ANALYST.

AUGUST, 1890.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS. ON DYED SUGAR.

BY CHARLES E. CASSAL.

(Read at Meeting, July, 1890.)

My attention has recently been directed to dyed sugar in consequence of a number of samples of moist sugar purchased in the ordinary way by the inspectors of one of the districts for which I am public analyst having been submitted to me. I am desirous of bringing the matter before the Society in its relation to the official work of public analysts, and of calling special attention to it as being one which appears to me to be of considerable importance, affecting general public interests as well as those of a large trade. Some two or three years ago it became known to many of us, and to persons in the trade, that certain firms had introduced sugar-crystals which had been artificially coloured. If I am correctly informed, it was not at first known in the trade that most, if not all, the sugar that was so introduced was beet sugar and not cane sugar; and I believe I am right in stating that the original name given to these products was that of *yellow cane crystals*. The present recognised trade name is "yellow crystals," and I do not suppose it will be denied, that the greater portion of the yellow crystals of commerce consist of artificially-coloured beet sugar.

The different samples submitted to me officially were plainly not the products of the same process of treatment, and therefore probably not of the same firm. They consisted of sugar crystals possessed of various shades of golden-yellow, approximating in some cases to orange. Since examining these samples I have obtained authentic specimens

of the dyed sugars of different firms, and have been engaged for some time in experimenting upon them. Somewhat varying degrees of skill would seem to be employed in the preparation of the sugars. Some samples have been very skilfully coloured, while with others the work has been coarsely and roughly done. It will be found by examination with a lens that in the products of certain firms a number of crystals are entirely colourless and can easily be picked out, while with others the uniformity of colouring is far more perfect. In addition, large nodules of a bright orange colour are frequently to be found in what, I presume, may be called the more inferior products.

The particular sugar to which my attention was first directed was one the dye on which can be detected with the greatest ease. The sugar itself, or a solution in water, strikes a peculiar purple-pink colour on the addition of a few drops of concentrated hydrochloric acid. The colour is characteristic, and, if the dye is present in unusually small amount, is best seen when about 25 to 30 grammes of the sugar are just moistened with hydrochloric acid in a deep conical porcelain dish. On standing, the mixture becomes of a dark, reddish-brown colour. Acetic acid has a hardly-perceptible effect, but appears to slightly intensify the orange colour of the crystals. Sulphuric acid produces the same effect as hydrochloric acid, but the subsequent changes of colour are not properly perceptible owing to the charring that takes place. Although from the point of view of the public analyst it is a matter of little or no immediate importance to be able to detect the particular dye-stuff or dye-stuffs which have been used, so long as an artificial dye can be certified to be present, I may direct your attention to the fact that these reactions are very similar to those obtained with certain of the so-called Tropæolins, especially in some respects with those which seem to be known in commerce as "Tropæolin OOO" or "Orange I.," and "Tropæolin OO" or "Orange IV." Tropæolin OOO is the only one which I have examined which gives the rapid change of colour from purple-pink to dark-reddish brown when treated with hydrochloric acid; which change, as previously stated, was obtained with the samples of sugar now referred to. I have not, however, succeeded in satisfactorily dyeing colourless sugar-crystals with this substance by itself, the colour obtained being dissimilar from that upon the commercial sugar. Further, acetic acid, when added to solutions of Orange I. and Orange IV., does not produce any red or pink colour, but merely what appears to be an intensification of the orange colour. For these and other reasons, which I propose to communicate later, I considered that while the particular sugars referred to had probably been dyed with Tropæolins, it was likely that a mixture of these bodies had been used. I have now reason to believe, from information which has been afforded me, that this is what in reality had been done. It must be remembered that great confusion exists with respect to the application of these commercial names. My remarks here apply to substances which have been purchased in the ordinary way under these names.

Other dye-stuffs are, however, used for the purpose of colouring sugar-crystals, which do not give the reactions with hydrochloric acid, and it has therefore been necessary to devise means for showing their presence with sufficient reliability to admit of their being certified to. By applying the following simple process, I believe that any of the dyed sugars at present on the market can be sharply distinguished from those sugars which contain no colouring-matter foreign to sugar. About 100 grammes in most cases (less can

be used if absolutely necessary), of the sample is washed in a flask with alcohol of 90° per cent.. This removes the dye in the majority of the cases which have come under my observation. In some cases several washings are necessary, in others one only. Treatment with alcohol of this strength removes the whole of the dye, leaving perfectly colourless crystals. I have found that it is advisable to use weaker alcohol with some dyed sugars, viz., from 75 per cent. to 80 per cent. The solution is filtered from the sugar, evaporated to dryness, again taken up with alcohol, and a skein of silk or wool (preferably slightly mordanted with aluminium acetate) treated with the solution, warmed for some time in the water bath, and subsequently well washed with water. The skeins are permanently dyed of a more or less marked yellow colour if a dye has been extracted by the alcohol from the sugar.

A sample containing only such colouring-matter as is natural to sugar, even by repeated washings with alcohol of 90 per cent., does not leave absolutely colourless crystals, and does not give a solution capable of permanently dyeing silk or wool.

One of the substances which has evidently been very largely used for the dyeing of sugar is the so-called phosphine or chrysaniline. Sugar dyed with phosphine can at once be detected by the foregoing process. As commercial phosphine is usually the nitrate or the hydrochloride of chrysaniline, no doubt it is one of these bodies that is used. Fibres dyed with phosphine are turned to a very characteristic pale greenish yellow by alkalis which is more intense than the colour on the fibres when the latter is slight, and acids slightly redden them. When the amount of the dye upon the fibres is slight, the application of alkali serves to "bring out" the colour. I have obtained these reactions in every case upon fibres dyed with solutions obtained from sugar suspected of being dyed with phosphine. Fibres dyed with extracts containing the colouring-matter which, for the sake of convenience, I will call the "Tropæolin dye," are reddened by mineral acids.

It is evident that great quantities of dyed sugar are sold in London and elsewhere. It is very generally sold as Demerara sugar, and not as "yellow crystals." Out of fifteen samples of sugar purchased recently in one of my districts as "Demerara," eleven were unquestionably dyed, and the adulteration was detected easily in each case by the processes previously described.

I may be allowed to quote here an extract from a report which I made upon the subject a short time back. "Purchasers are under the impression that they are being supplied with genuine cane sugar (such as "Demerara"), whereas they are in reality getting sugar crystals which have been artificially dyed. In this way beet-root sugar can be coloured and sold as "Demerara," and it is generally admitted that weight for weight, under ordinary conditions of use, beet-root sugar does not give the same "sweetening" as cane sugar. A natural product having been tampered with by admixture with an artificial dye, whatever the real object may have been in the preparation of these sugars, it is obvious that the proceeding enables one article to be substituted for another." I am reliably informed that when "Demerara" is asked for on the market, cane sugar from Demerara is expected. The market price further, is stated to be from 17s. to 18s. per cwt. for Demerara, and for "yellow crystals" from 12s. to 13s. per cwt. A somewhat remarkable statement has been made with reference to my intention of certifying

artificially dyed sugar sold as Demerara as adulterated ; namely, that cane sugar produced in the West Indies is dyed there with the same substances as those previously alluded to. This is merely an attempt to create a new definition for Demerara sugar. It might almost as logically be urged that if growers of coffee were to take to mixing chicory with their product before exportation, we should no longer be able to regard chicory in coffee as an adulterant. If the case is not strictly parallel the device is the same. It may, on the other hand, justifiably be contended that the sugars dyed as I have described are adulterated, for they can be certified to have been produced by dyeing colourless sugar crystals yellow, with an artificial organic colouring matter not natural to sugar.

Another aspect of the subject in reference to which I may again quote from my report is this : " Many substances used as dyes, and closely related to the classes to which those found belong, are known to be of a poisonous nature, while the action of others, when ingested, is unknown, while others again are asserted upon very insufficient and unsatisfactory evidence to be non-poisonous. Apart, therefore, from other considerations, these cases afford an illustration of the impropriety of tampering in such a way with articles of food without, at least, an open statement of what has been done. Although in dyeing an article of food the weight of the dye-stuff used must be exceedingly small as compared with the weight of the substance dyed, it must be admitted that the proper course of action in the present state of knowledge on the subject, is, as far as possible, to prevent the use of dye-stuffs of the general character indicated for colouring articles of food, if for no other reason, in view of probable and perhaps dangerous abuse."

In giving official certificates in these cases it will be tolerably obvious that it is undesirable, as well as unnecessary, to make too specific statements as to the precise nature of the dye or dyes that may have been used. The statements that may be made by experts in giving evidence in matters of this kind very largely depend upon the standpoint from which they are viewed. If, for instance, a definite statement were made as to the precise character of a complicated dye-stuff present in very small quantity in sugar, or anything else, it would no doubt be perfectly easy and conscientiously justifiable for the gentleman who had prepared the mixture to swear in the witness-box that the compound certified was not present at all, if the compounds were not in every respect, physical as well as chemical, in composition, as well as in constitution, identically the same in the opinion of every chemist of repute, or even of non-repute. Whatever the standpoint of the chemist consulted by the manufacturer may be, and I fully admit the propriety and value of such consultation, when professionally legitimate and publicly useful, the position of the public analyst is clearly defined. It is his duty to the community which he is appointed to serve to advise them of the existence of adulteration when it comes before him, and to do so without fear and without favour. That the dyeing of sugar in the manner indicated, and its sale as genuine natural sugar, and the application of the process for the purpose of enabling one thing to be palmed off for another, are adulterations, and, therefore, offences against the law, I, for one, have not the faintest doubt. The public have every right to expect that natural food products shall not be coloured with artificial dyes without an open statement of the fact. I desire to express my thanks to my assistant, Mr. Gerrans, A.I.C., for the help which he has afforded me in carrying out the necessary experiments.

DISCUSSION.

DR. DUPRE said he had repeatedly had sugars submitted to him artificially coloured, but never as public analyst, and he should feel doubtful himself as to certifying sugar as adulterated, simply because it was artificially coloured, unless it was proved that the colouration was for the purpose of deceiving and enabling it to be sold as Demerara sugar. He would like to hear the opinions of other analysts as to whether the mere fact of sugar being coloured was to be taken as an adulteration. Butter was often coloured, so was milk, but many analysts do not consider the colouration of butter to be an adulteration. It had been a long-continued custom, and the public liked to see their butter yellow. If an artificial butter was coloured with a view to its being passed off as a genuine butter that was a different matter, but the mere fact of its being coloured was not an adulteration.

He quite agreed with Mr. Cassal that a public analyst was not bound to state the exact composition and character of what he supposed to be the adulterant, for if it came to that, and they should have to put the exact composition of such and such a substance, and if they linked together wrongly the oxygen and hydrogen, and were then liable to have their certificates set aside, the sooner they gave up the better. All that the public analyst could be expected to do was to say that this particular sample, say of sugar, is artificially coloured. If he can go a step farther and say that the colouring matter is so and so, that would be an advantage, but otherwise it would be a most dangerous thing for the public analyst to bind himself down to a particular composition. He did not think that the Act at all required the public analyst to do that.

He would like to ask whether Mr. Cassal had submitted these colouring matters to spectroscopic examination, because so many of them could be identified with a tolerable degree of certainty by means of the spectroscope.

He had also found that artificial colouring matter added to sugar was almost as easily distinguished by the action of water, natural sugars colouring water very slightly only, whereas artificially-coloured sugars imparted a marked colour, like chicory compared to coffee.

The colouring matter added might be the same kind as that of the natural sugar, but there was this striking difference—when water was added to the sugar the artificial colouring matter was dissolved off readily and left colourless crystals, the natural sugar remained the same colour.

MR. A. W. STOKES said that the public analysts were on the horns of a dilemma. It is well-known that almost all Demerara sugars contain about one-third of a grain of chloride of tin per lb., while most other sugars were artificially coloured with aniline colours in minute quantities. So that, from Mr. Cassal's view of the subject, few moist sugars would pass as unadulterated.

Mr. Stokes gave the opinion of Mr. F. J. Scard, the analyst at Demerara of the Colonial Company, who contends that the sale of fictitious "Demerara sugar" should be stopped under the Merchandise Marks Act. Mr. Scard holds that Demerara sugar keeps at the head of the market because of its retaining the natural aroma, tint, and flavour of the sugar-cane. Over-refining gets rid of these, and produces a less palatable article. He contends that chloride of tin is used merely as a mordant to fix these natural qualities; its use could be dispensed with, though not at present conveniently.

MR. ALLEN said he thought they ought to distinguish between their present position and the position they might be in in the future. Dr. Dupré had raised the question how far they could obtain convictions under the Sale of Food and Drugs Acts in cases of mere colouring, provided that the colouring matter was harmless; but Mr. Cassal and the speaker had in recent papers foreshadowed the time when the present

law might be amended; and the more instances they could bring before the notice of the authorities, and the more facts they had to go on, the stronger would be their position whenever the time came for fresh legislation. It was, of course, very desirable where possible to positively identify the nature of the colouring matter used, but that was by no means a necessity, any more than it was necessary for them to specify the exact nature of the fatty matters added to butter, or than it would be to specify the exact nature of hop substitutes supposing that any legislation were to be founded on the investigation of the recent committee. Mr. Allen said he certainly objected to have anything put into sugar for the sake of colouring it and making it pass as Demerara sugar. It was possible that such an addition might be dealt with under the Trades' Marks Act, but it appeared to him that the two facts taken together, namely, that it was not Demerara sugar, and that the colouring was put in for the purpose of deceiving, would make a good case to go before a magistrate.

MR. B. E. R. NEWLANDS said it would be a very extraordinary thing to find a sample of sugar that was not coloured. All sugars, such as loaf, granulated, crystals, and yellow crystals were coloured by artificial means, either with a blue or yellow colouring agent, and if the vendors of one kind of sugar were to be punished for introducing a harmless colouring matter, then those of all other kinds of sugar would be equally liable to prosecution.

In the West Indies yellow crystals were produced by the action of sulphuric acid or of stannous chloride, or, as at home, by the addition of a colouring matter. In the former cases, two or three gallons of the agent were added to a panful of sugar, and what Mr. Cassal would call a natural colour was produced. A good deal had been said as to palming off refined yellow crystals as Demerara, but if they referred to to-day's papers, they would find that, in many cases, the price of Demerara was lower than that of yellow crystals, the former selling at from 15s. to 16s. and the latter from 16s. 3d. to 17s. 3d. per cwt. It is, therefore, evident that the latter realise a higher price than the sugars they are supposed to imitate.

In Mauritius, Demerara, and the West Indian Islands, sugar, in addition to being coloured by sulphuric acid or stannous chloride, was coloured in exactly the same way as by refiners in this country, and therefore it would be impossible by any process of analysis to decide as to its place of origin.

What useful object could be attained by prosecuting grocers for selling yellow sugar, beyond providing work for the analyst? They might as well prosecute people for selling coloured sugar candy or Chelsea buns, or anything else coloured by artificial dyes. Dr. Dupré had suggested that the presence of artificial colouring matter in a sample of sugar might be ascertained by washing it with water. It was, however, extremely difficult to find a sample of sugar from which the colour could not be removed by water; he had been promised such a sample as a curiosity, but it had not yet arrived.

In turning out a standard article, such as yellow crystals, it was necessary to keep the colour perfectly uniform, and the refiner, therefore, had to add a minute amount of colouring matter, not exceeding one part in fifty thousand, for this purpose.

Manufacturers of sugar in the West Indies had often occasion to colour their products in the same way, and the sample of so-called "genuine Demerara sugar" handed round by Mr. Cassal, was thus artificially coloured. Whilst defending the sale of yellow crystals, he of course agreed that it was highly improper to describe them as "Demerara" unless they were actually produced in that colony.

MR. HEHNER said, that inasmuch as several aniline colours were of a decidedly poisonous nature, it could not be denied that the public analyst had the right and the duty to concern himself with the question of dyed sugar, and that he should know the

exact nature of the colouring matters employed. He had no grounds for believing that injurious colours were used in this particular case, but just as a public analyst might not to know all details of technical and manufactory processes, so manufacturing chemists might possibly be ignorant of the physiological action of the colours which they added to food materials. As instances, Mr. Hehner mentioned Dinitro cresol (also known as Victoria yellow and under other names) and Dinitronaphthol (Martins yellow) which were both eminently poisonous.

MR. HERON said he might say at once with regard to what Mr. Hehner said, that no colouring matters were used for colouring sugars which were of a poisonous nature; concerning the point brought forward by Mr. Stokes as to the use of chloride of tin in the West Indian plantations, such was not the case. As a matter of fact the chloride of tin was used there for the purpose of destroying the dark colouring matter present in the crudely prepared raw sugar and imparting to it a bright golden yellow colour so characteristic of Demerara sugar, and thereby making an inferior class of sugar appear of a better quality than it really was, and, as proof of his statement, he would ask any gentleman present, who was at all interested in this matter, to make a complete analysis of any West Indian sugar and compare it with an analysis of a home-refined sugar sold on the market as yellow crystal, and it would readily be seen that the latter was the purer of the two. He maintained that the colouring matter referred to by Mr. Cassal was not added to the sugar by refiners to give it a fictitious value, but merely to meet the public taste, which at present seemed to run in that direction, that this colouring matter was perfectly harmless to human beings and animals, and that the sugar sold as yellow crystal was in his opinion much purer and better in every way than any foreign sugar of a similar character at present on the market.

DR. DUPRE said that they as a scientific body should not accept the assertion of anyone, whoever he might be—let sugar manufacturers assert ever so much that the colouring matter they added was innocent—he should not pay the slightest regard to it unless they were prepared to state the nature of the colouring matter added, and then let him judge whether or not it was injurious to health. Did Mr. Newlands really mean to assert that all their sugar was artificially coloured? He was under the impression that some years ago the—what they call natural—colouring matter of sugar was a kind of caramel, and he should be exceedingly astonished if Mr. Newlands told them that our old-fashioned sugar-candy was coloured with chloride of tin or sulphuric acid.

That colouring was what he called natural which remained in the sugar when treated with water. Mr. Newlands said there was no sugar now in the market the colour of which could not be washed out by water; that was to say that no sugar manufacturers really gave them unadulterated sugar.

MR. B. NEWLANDS said that the lower qualities of sugar-candy did contain the natural colouring matters of sugar, viz., caramel and dirt. The better qualities were produced from raw sugar which had been coloured by the agency of sulphuric acid, stannous chloride, etc.

MR. HEHNER said he would desire to protest against the often exploded doctrine that we should in any way be guided by the popular wish in food matters. The popular wish had been used by sophisticators for many years past as an excuse for every abomination perpetrated upon an ignorant public; no kind of adulteration was ever carried out without its being alleged to be in obedience to the popular wish. Years ago pickles and preserved peas were sold as green as the leaves on the tree, coloured with copper, and bloater-paste made red with brick dust; nowadays mustard and cocoa was mixed with flour and starch—all mixed by the obeying manufacturer to meet the popular wish. The public analyst, however, had to disregard the popular wish and raise the standard of

purity of food, if necessary, *against* the popular wish. Was not even milk mixed with water because the people desired to have milk cheaper than it could possibly be produced by legitimate means? He hoped that public analysts would not allow themselves to be carried away by such phrases as "the popular wish."

MR. CASSAL, in reply, said he was very strongly of opinion that a public analyst was not bound to state the exact nature of an adulterant. He would go further, and say that it was always undesirable to enter into technical details of any kind before magistrates and others, who were utterly innocent of any scientific training, and therefore quite incapable of judging of the merits of the most elementary scientific questions.

Replying to Dr. Dupré, he stated that he had not as yet applied the spectroscope to attempt to identify the colouring matters, but that he proposed to do so. He desired to express his thanks as a public analyst to Mr. Newlands and to Mr. Heron for the opinions they had expressed and the admissions they had made. He was sure that the members of the Society generally would join with him in doing this, more especially those who occupied official positions as public analysts. At the same time he could not congratulate them upon the defence which they had endeavoured to make of the practice of colouring sugar with artificial dyes. He noted, with much satisfaction, that they did not agree with each other as fully as might have been expected, and that certain of the remarks which they had made were surprisingly contradictory. With reference to chloride of tin, for instance, the Society had been informed that it was used as a "mordant" to "fix" the "natural aroma, tint, and flavour of the sugar-cane," then that it was applied for the purpose of producing colour in colourless crystals; and, finally, "as a matter of fact," that it was used for destroying the dark colouring-matter of raw sugar. These statements appeared to require some revision by those who were interested in sugar manufacture, and he would leave the matter in their hands. It led him, however, to point out the necessity of distinguishing between sugars artificially coloured by producing a "natural" colour in the form of caramel, with such agents as chloride of tin or sulphuric acid, and dyeing with aniline dyes for a purpose which in all probability was very different. Mr. Newlands had told them that all sugars were coloured, and had referred to the use of blue for loaf sugar. It was hardly necessary to point out that this was an altogether different thing—it did not serve the purposes of deception. They had also been told about the prices, and he (Mr. Cassal) had been duly rated for meddling with a question which ought to be left to the superior ability of the "sugar expert," but he had also had the opportunity of consulting authorities even as great, perhaps, as those present, and the opinions they expressed were different upon both points.

It having been freely admitted that the dyed crystals were beet-root sugar, and that hardly anything else could be obtained on the market, he was glad to find Mr. Newlands agreeing with him that to sell "yellow crystals" as "Demerara" (which word implied *cane-sugar*) was highly improper unless they had been produced in that colony. In other words, beet-sugar should not be sold as cane-sugar, and anything which facilitated such a sale was therefore condemnable. In this connection he would remind members that of fifteen samples of sugar recently purchased as "Demerara" in Kensington, eleven consisted of dyed crystals.

What was the object of dyeing colourless sugar-crystals with an aniline dye or dyes? Mr. Newlands and his friends would not go so far as to deny that the dyes used were aniline derivatives of some kind. It was said that it was for the purpose of maintaining continuity of trade and of pleasing the public taste.

There was possibly some validity about the trade continuity argument, and he should be happy if he could bring himself to think that this was indeed the reason.

Butter and milk had been mentioned, and perhaps the continuity argument might apply in a limited sense to butter ; he could not admit it for milk, and in reference to all food-products, colouring practices required to be sharply looked after. The object of most of them, if not all, was to produce a false impression on the mind of the customer ; and, as had been pointed out, they were fraught with danger. The only satisfactory method was to treat the matter rigidly, and to make it compulsory upon all vendors of food containing extraneous products to state what they had done. The "public taste" excuse for adulteration had been fully dealt with by Mr. Hehner ; it was a very ancient excuse, and nothing more need be said about it.

They had been told that all the colours used, or likely to be used, were absolutely harmless in any amount. That was a somewhat bold statement, and he (Mr. Cassal) fully agreed with what Dr. Dupré had said about it. Something more was wanted than the mere statement of a particular person or firm that what they in their wisdom chose to put into food was "perfectly harmless" on the strength of some nebulous experiments made by somebody—they knew not whom—somewhere.

The samples moistened with acid which he had passed round were intended, as he had said, to show the difference between a sugar which would give the peculiar pink colour with hydrochloric acid, and one which would not. He thought that he had made it sufficiently plain in his paper that he relied upon the removal of the dyes by suitable solvents, and their purification if necessary ; and not simply upon the incident that some of these dyes could be detected at once by the addition of a mineral acid.

The attempt alluded to in the paper to prevent the maintenance of the definition which could at present be given to Demerara sugar by stating that cane-sugar produced in Demerara was there coloured with aniline dyes and then exported was certainly ingenious, and reflected great credit upon whoever had evolved the idea ; but it was singular that those who believed in this theory should admit that nearly all, if not all, the dyed sugar on the market was beet. Even if it was admitted that dyes were applied to cane-sugar in Demerara, he contended that the case was not affected. It was simply adulteration in Demerara instead of in England. By Demerara sugar was understood cane-sugar, and cane-sugar containing only such colouring matter as was natural to sugar.

He thought that the Merchandise Marks Act could certainly be applied to the case, and if it were so applied some of the vendors of "crystals" or "Demerara" might be disagreeably surprised. The penalties were not likely to be of the trumpery kind to which they were accustomed under the present Adulteration Acts. He was much gratified to find that the general consensus of opinion in the meeting was in favour of the views which he had put forward.

(Conclusion of the Society's Proceedings.)
