STUDIES ON CIDER AND PERRY. No. I. SULPHITE PRESERVATIVES.

BY HERBERT E. DURHAM, M.A., M.B., B.C. (CANTAB.), F.R.C.S. (ENG.), *Hereford*.

THE practice of using preservatives in the process of making cider is very widely spread, and moreover is much encouraged by the makers of the preservatives.

There are three chief reasons, or perhaps one should rather say excuses, for the use of preservatives; the first is to prevent the complete fermentation of the sugar in the must—in popular language "to preserve the sweets,"—the second to cure or prevent "diseases" of fermentation, and the third (when sulphites are used) to prevent the blackening which occurs with certain kinds of apple. The nature and cause of this blackening is not fully understood, and I hope in a further communication to throw some light on it.

It would appear that some years ago salicylic acid was the substance favoured by cider makers in this country, as benzoic acid (or its salts) is now in favour in the United States. From my analyses of representative samples of the products of the industry in this country, it seems that sulphite preparations are now more in vogue. In part, this change may be due to the attention which has been drawn to the presence of added salicylic acid in ciders, but perhaps it is still more due to the excuse that sulphurous acid is not "objectionable." Thus whilst some makers only go so far as to say that their products contain no objectionable preservative, at least one maker has not scrupled to state that his cider contains no added chemicals, although it contains a considerable percentage of sulphurous acid.

According to Laborde¹ and Warcollier², complete sterilisation of the must is only secured by the addition of 500 milligrams of sulphur

¹ Laborde, Cours d'Oenologie, Paris, 1908, p. 265.

² Warcollier, Le Cidre et le Poiré, 20^e Année, Paris, 1908, p. 202.

Journ. of Hyg. 1x

dioxide per litre; but smaller doses have more or less consideral inhibitory effect.

Pending the promised report of the Department of Agricultu Washington, U.S.A., there does not appear to be much direct expe ment on the deleterious action of sulphurous acid on man. Leuch (v_i Laborde) has shown from actual trials that a single dose of wine (300 c about 1 pint) containing 50 mgrms. of free, or 300 mgrms. of combin sulphurous acid per litre gave rise to discomfort; but the effect repeated doses does not appear to have been tried. These observation led to the assumption that "free" sulphurous acid was five times me deleterious than the "combined," and have led to legislation on t subject. It is interesting to observe that, according to Laborde, t first regulation of this antiseptic in France was undertaken on accou of heavily sulphited English beer which was imported into Pa Besides the deleterious action of the acid in undue quantity, the use this and other preservatives undoubtedly assists the manufacture fraudulent concoctions which masquerade under the title of cider or wi It may be concluded that the most recent French law on the limitati has been introduced to minimise such frauds, which have done so mu harm to the French wine industry; and it may be remarked that t law has reduced the permissible amount of sulphur dioxide to less th a third of that previously allowed.

Another reason against the use of sulphur dioxide is the effect flavour of the product. Warcollier (*loc. cit. supra*) says that 30mgrms. per litre are recognisable, if recently added to cider; but added to the must before fermentation considerably larger doses are 1 detected. It is worth while mentioning that some of the samples, wh I analysed, were submitted to a practised palate, and the prediction the presence of the acid was confirmed chemically, except when of about 10 mgrms. per litre were present.

There are, then, three lines of argument against the unlimited u of sulphur dioxide in cider—detriment to health, assistance to fraudule concoctions, and deterioration of flavour.

The retention of a portion of unfermented sugar in the finish product can be accomplished by the old process of repeatedly "rackin "racking" consists in drawing off the liquid from the sediment of yea and inasmuch as the yeasts are continually abstracting food materi (notably nitrogenous and phosphorised substances) from the must i: themselves, the racking impoverishes the liquid of these all-import constituents, until there is not sufficient to support the further grov of those yeasts which remained suspended in the fluid. The keeping quality of the cider then depends upon the lack of nutritive material combined with the preservative action of the carbonic acid gas and alcohol. It is worth noting here that in France the addition of "artificial" carbonic acid has to be declared.

In practice the effect of racking is often or even usually enhanced by using "matched" casks, that is to say the racked off liquid is received into casks in which a "match" of sulphur has been burnt. Thereby the mere mechanical action of the racking is aided to a more or less considerable degree by the antiseptic action of the sulphur dioxide. From this, the step to a deliberate addition of antiseptic (sulphurous acid, sulphites, salicylic acid, etc.) is a small one, especially as the labour involved is also lessened.

Another method of producing sugar-containing cider is the well known champagne process, in which the use of antiseptics is superfluous.

There is yet another method of obtaining a sweet, but not sugary, cider. This also does not involve the use of antiseptics for the sweetness is obtained by the use of artificial agents such as saccharin or dulcin. This process is still practised with apparent impunity in this country, but abroad, both in cider- and wine-making it is only carried on in defiance of the law. In this country there is a reprehensible laxity in regard to the use of these artificial sweetening agents, the use of which for these purposes is entirely fraudulent.

Laws concerning use of sulphites, etc., in food products.

(1) United Kingdom. In order to ascertain the state of the law in this country with regard to the use of sulphurous acid and sulphites, I enquired of my friend, Dr Buchanan, Inspector of Foods, Local Government Board. His reply dated 10/vii/1908 states that "in the United Kingdom there are no prescribed official limiting quantities of sulphurous acid or of sulphites permitted in foods or drinks."

Since there are no regulations, it is not surprising that an undue sense of security is given to the public by the recommendations or analyses of unofficial trading concerns. Thus I find that ciders have been given guarantees of "purity" although samples of the same make contain much sulphurous acid, in some cases more than the former French maximum of 200 milligrams per litre. Such guarantees of purity might perhaps be prevented if it were made obligatory to declare

2-2

the presence of preservatives not only on the package but also in the analytical report or certificate.

(2) France. The amount of sulphurous acid was first limited by law in France in 1901, the amount permitted in wines and cider being limited to 200 milligrams per litre, whether "free" or "combined." In 1907, the amount permitted was increased to 350 milligrams per litre, and it was also enacted that in cases where alkaline bisulphites were used, only 20 grams per hectolitre were allowed. This year, 1908, the permissible dose has again been altered, so that now only 100 milligrams of the acid (free or combined) per litre or 10 grams of the salts per hectolitre are allowed.

(3) United States of America¹. There are special State Regulations and also Federal Rules; these latter apply only to interstate and export commerce.

The different States have various regulations; thus "several States, e.g. Wyoming, Indiana, North Carolina, North and South Dakota, Texas and Wisconsin prohibit the addition of sulphurous acid or compounds derived therefrom in foods and beverages." The Federal rules limited (see Food Inspection Decision 76, issued July 13, 1907) the amount of sulphur dioxide, subject to declaration, to 350 milligrams per litre provided that not more than 70 milligrams are in a free state. It was recognised that some regulation was needed, as is shown by the following extract (p. 9). "It is absolutely necessary to restrict in some manner the sulphur dioxid in cases in which it is used under conditions, such that it may enter into combination with acetaldehyde, sugars, etc., present in food products, and it is believed that under the restrictions suggested, the public will be protected from products unduly sulphured, during the period which must elapse before experimental evidence can be obtained, as to whether a total restriction in the use of sulphur dioxid, under all the conditions mentioned, is necessary on account of the toxic properties possessed by sulphur dioxid in the combined form."

It may be presumed that the limitation by this regulation was designed to permit the importation of French wines. According to rumour, difficulty arose through importation of more highly sulphited products than this decision allowed, and an amended decision is now in force (F.I.D. 89, issued March, 1908). So that pending determination by the Referee Board of consulting scientific experts, "no objection will

¹ I am indebted to Dr Buchanan of the Local Government Board and to Dr Wiley, Chief of the Bureau of Chemistry of the United States Department of Agriculture, for the information concerning the law in U.S.A. be made to foods which contain the ordinary quantities of sulphur dioxid, if the fact that such foods have been so prepared is plainly stated on the label of each package." It may be noted here that benzoate of soda appears to be the favourite (or even "necessary") adulterant for cider; the limit allowed, with declaration, being $0.1 \, ^{\circ}/_{0}$. It is further provided (F.I.D. 76, p. 9) that foods containing such declared additions shall not be labelled as "guaranteed to conform with the Food and Drugs Acts."

(4) Austria-Hungary was the first country to prescribe limits, and in 1885 a limit of 20 milligrams was laid down; later it was increased to 80 mgrms. Italy, Belgium, and the Argentine Republic may be mentioned as other countries which have direct laws on the subject.

Free and combined sulphurous acid.

It has already been mentioned that sulphurous acid is recognised to take two forms in substances like wines, the "free" and the "combined."

The basis of this distinction appears to be that combinations of the acid which are not attacked by iodine are called "combined." To some extent this distinction is probably academical, since exposure to the free hydrochloric acid of the gastric juice would decompose the so-called "combined" acid.

The mode of estimating the "free" sulphurous acid in wines consists in a rapid direct titration of the acidified wine with standardised iodine solution and starch indicator, the operation being conducted under an atmosphere of carbonic acid gas¹.

Dr Wiley informs me that direct titration with iodine is the official method in U.S.A.

Direct titration with iodine.

- 1. Dry cider free from SO_2 required addition of iodine equal to $0.166 \, {}^{0}/_{0} \, SO_2$.
- 2. Dry cider ditto ditto $0.014 \circ/_0 SO_2$.
- 3. Same sample after treatment with animal charcoal, ditto 0.003 $0/_0$ SO $_2$.

It has been pointed out by Matthieu² that there are many substances in wines which are capable of taking up iodine besides any SO_2 which may be present, and that, so far as this adulteration is concerned, the

¹ Lunge, Chemisch-technische Untersuchungsmethoden, 111. p. 622, Berlin, 1905.

² Quoted by Dujardin and Salleron, Instruments de précision appliqués à l'oenologie, 4th ed., p. 222. Paris. method is quite fallacious. The same objection holds good for cider, as is well shown by some titrations which were made on samples in the manufacture of which no sulphuring whatever had been employed, and in which no appreciable amount of SO_2 was revealed by the distillation method.

Animal charcoal causes a very complete removal of the tannins and other reducing matters in the cider, and the effect upon the amount of iodine required is marked. It is possible that absorbed oxygen might claim some of the action of the charcoal.

One further trial was made upon the liquid of a pure culture of yeast in a sugar-salts solution, which had fermented out to "dryness." In this case the amount of iodine required was equivalent of $0.0128 \,^{\circ}/_{\circ}$ SO₂, which shows perhaps more strikingly the uselessness of the method.

Combined sulphurous acid is estimated by subtracting the amount of "free" from the "total" sulphurous acid; this latter is found by a distillation process which is dealt with below under the heading "methods."

According to Kerp (quoted by Lunge, p. 623) a portion of the SO_2 added to wines becomes oxidised to sulphuric acid, but the greater part combines with aldehydes, sugar, etc., so firmly that iodine is no longer able to destroy and oxidise the combination. This cannot be said to be true of sound cider, in which the reducing substances aid in sheltering the SO₂ from oxidation. It may be added that iodine seems to be able to oxidise all the SO₂ provided that time is allowed.

This definition of "free" acid does not seem to be of great value, and I think that such portion of the acid as might come off with a plain distillation process might be more worthy of the title "free sulphurous acid."

The following two experiments were made in order to see what proportion of the total sulphur dioxide would distil off from sulphited cider without any addition of mineral acid to liberate it.

The results obtained were :---

```
Sample A.

Distilled without any addition gave BaSO_4 0.0742 \text{ g.} = 0.02036 \text{ g.} SO_2 0_0^{\circ}.

Distilled with P_2O_5 gave BaSO_4 0.0809 \text{ g.} = 0.0222 \text{ g.} SO_2 0_0^{\circ}.

Sample B.

Distilled without any addition gave BaSO_4 0.0961 \text{ g.} = 0.0527 \text{ g.} SO_2 0_0^{\circ}.

Distilled without any addition gave BaSO_4 0.0961 \text{ g.} = 0.0527 \text{ g.} SO_2 0_0^{\circ}.

Distilled with P_2O_5 gave BaSO_4 0.01115 \text{ g.} = 0.06068 \text{ g.} SO_2 0_0^{\circ}.

Direct titration with iodine gave equivalent of 0.0224 \text{ g.} SO_2 0_0^{\circ}.
```

 $\mathbf{22}$

These samples gave contained sulphuric acid (SO_3) 0.0068 and 0.0087 g. per 100 c.c. respectively.

It will be seen that a very large proportion of the SO_2 was sufficiently free and volatile to pass over without the liberating action of the strong mineral acid; moreover, as will be shown hereafter, the sulphuric acid is within normal limits, so that no considerable amount of oxidation can have occurred. Inasmuch as there was a possibility that some amount of oxidation might have taken place in samples, especially if acetification took place during manufacture, as a routine, I took estimates of the sulphuric, as well as of the sulphurous acid in all the samples which were tested.

It may be concluded that the term "free" sulphurous acid in ciders might well be defined as the amount which is sufficiently free to pass over on distillation when no liberating acid is used.

Methods.

The simplest mode of revealing the presence of some preservative in cider, is to place a portion of the sample to be tested in a sterilised plugged tube and inoculate it with a pure vigorous culture of cider yeast. If necessary sugar may be added to make the proportion about 3 or 4 per cent. Inasmuch as there may be a poor supply of nitrogenous food in the cider it may be desirable to add a small proportion of an ammonium salt, such as the phosphate, but not more than 2-3 milligrams per 100 c.c. Many samples of cider, which are really free from antiseptic additions, do not require this addition of nitrogenous and phosphatic food and probably it is unnecessary in any case, if thoroughly well-nourished yeast culture is used. It must be remembered that if too much help be given to the yeasts, they will more readily and rapidly As a routine, I overcome small proportions of inhibitory substances. put up two lots, one with the addition of the ammonic salt and the other without. In some two to four days the effervescence of active fermentations should have set in, if it do not the presence of some preservative may be suspected.

In the case of heavily sulphured or more correctly "sulphited" ciders, it will be seen that the yeast does grow to some extent in the form of flocculi and without production of turbidity at first; but after a more or less prolonged interval the sulphites become oxidised to sulphates and the yeasts become enabled to attack the sugar in the usual way. In one case, where a nearly full pint bottle of a sulphited product was inoculated and plugged with cotton wool, the delay in oxidation, owing to the small amount of air contact, was such that the amount of sugar remained unaltered for three months; eventually the sugar fermented out and all the sulphurous acid had been oxidised to sulphate.

Whilst this fermentation test only indicates the presence of some preservative or other in general, yet when the antiseptic action disappears spontaneously there is a presumption in favour of the employment of sulphite.

The samples were obtained in the open market, sometimes by the kindness of friends. I am also indebted to Mr Barker, Director of the National Fruit and Cider Institute, for two samples made at the Institute.

Sulphurous acid. The determination of the "total" sulphurous acid was made as given by Lunge (op. cit. p. 621); namely CO_2 was run through the apparatus, 100 c.c. of the freshly opened sample were run in, and 5 c.c. of syrupy phosphoric acid added. The CO_2 current was allowed to slacken off till towards the end of the distillation, for the samples usually contained enough to give a fair current. An upright glass worm condenser was used, and led into a "Peligot" (triple bulb U-tube) tube containing the iodine solution; I found it advantageous and economical only to introduce about 10 c.c. of the latter solution at first and then add more from time to time if the colour showed signs of disappearance, thereby a rough idea of the amount of sulphurous acid may be formed and a waste of the iodine solution avoided. The absence of sulphate in the iodine and other reagents was of course determined. Finally the SO_2 in the distillate was determined gravimetrically as barium sulphate.

In order to avoid the tedious gravimetric process, some attempts were made to titrate the iodised distillate directly with thiosulphate; but it was found that the results were not satisfactory, probably because some bodies are carried over in the distillation which are capable of combining with the iodine. For approximate results Dr Bigelow informs me that this process is sometimes employed in the Department of Agriculture, U.S.A., but errors are considered to arise from volatilisation of part of the iodine.

A mode of attacking the question of the differentiation of "free" and combined acid, which I tried, may be mentioned. This was by attempting fractional precipitation of barium sulphate in the course of direct iodine titrations, with subsequent additions of iodine; two circumstances, however, made the attempt useless, for the fineness of the precipitate caused too much loss through the filter, and the difficulty of avoiding all air contact during filtration was too great.

Sulphuric acid. The determination of the sulphuric acid demands a few remarks for the standard method (vide Lunge op. cit. p. 605) leaves something to be desired. The sample, acidified with HCl, is thoroughly boiled and BaCl₂ (about 2 c.c. of $10^{\circ}/_{\circ}$ solution) is added; if, then, the precipitate is filtered off, washed and merely incinerated, as is advised, an appreciable loss of BaSO₄ is often entailed because the precipitate comes down rather too fine in the presence of the sugar, etc., of the cider to be well retained by the "baryta" filter paper; it is, however, retained by virtue of organic matters which are precipitated with it. On washing with water, and still more with alcohol, much of this organic precipitate can be removed, but then $BaSO_4$ in appreciable amount also passes away. On the other hand, when the incinerated precipitate is treated with sulphuric acid to decompose carbonate, there may be a gain of BaSO₄, if the washing process has not been sufficiently thorough to remove all barium compounds other than the sulphate.

In order to avoid these sources of fallacy the following process was adopted. When a perfectly bright filtrate was obtained, a slight washing with water was given, the filtrate being collected in a clean flask to see that no BaSO₄ was lost. The paper was then allowed to drain and nearly dry spontaneously. It was then cut into three by cuts concentric with the paper. First the point of the cone (containing the greater part of the sulphate) was dabbed carefully into molten fusion mixture (KNO₃ and Na₂CO₃), the next zone was similarly treated; the outer zone was sometimes burnt directly and added to the fusion.

In this way the organic impurities are rapidly oxidised and the sulphate remains uninjured; strictly speaking, the result obtained is the "total sulphur" in the paper and precipitate, but, in the case of yeast-free cider, the margin of inaccuracy is very negligible. If damp, the paper and precipitate gently fizz in the fusion mixture without spluttering or inflaming, if a slight amount of care in dabbing is used; if the paper is quite dry great care must be taken or the paper will blaze and carbonisation will occur, it is best to remove the gas burner whilst the paper is dabbed.

The fusion mass is then taken up with HCl and water, freed from nitrate, the resulting BaSO₄ purified with successive lots of HCl, and finally weighed. I find that some iron compound is always carried down with the barium salt and is not easily removed completely.

Mode of addition of sulphites.

The French law lays down that the sulphurous acid shall be pure, and proceed from the combustion of sulphur; further, that where alkaline bisulphites are used they shall be pure and crystalline.

Sulphiting may be performed :---

- (1) By the fumes from burning sulphur.
- (2) By blowing in SO_2 from a container filled with the liquefied gas.
- (3) By adding sulphites of the alkalis.

(1) Fumes of burning sulphur. The primitive and much practised method of "matching" casks, i.e. burning a strip or "match" of canvas or paper impregnated with sulphur, has already been mentioned¹.

(2) Use of liquefied sulphurous acid. Capsules and cylinders of liquefied sulphurous acid are to be obtained in the market and also arrangements for liberating the desired dose.

Impure solution of the acid can also be cheaply obtained. To what extent either of these products are employed in this country I have no knowledge.

(3) Use of alkaline bisulphites. The metabisulphite of potassium seems to be the most favoured salt, especially in France, owing to its high proportion of available SO₂, (K₂S₂O₅ = 57.6 °/₀ SO₂). The sodium salts are said not to be much used.

The calcium sulphites are much used by British brewers for adulterating their beer, and it would appear that they are also employed by cider-makers; for, there is a much advertised substance under the name of "Ham's improved antiferment to preserve the sweets of cider." I obtained a sample of this product and found on analysis, that in its natural air-dried condition it contained :—

$$SO_2 = 39 \, {}^0/_0$$

 $Ca = 26.44 \, {}^0/_0$ (CaSO₃, $2H_2O = SO_2 \, 40.9 \, {}^0/_0$
Ca 25.67 ${}^0/_0$).

According to the instructions, the contents of the packet were to be added to 100 gallons of cider, and if this was not efficacious, a further

¹ According to a legend related by Mr Ozzard at the National Fruit and Cider Institute, Long Ashton, Bristol, an abbot who had trouble with his cider called in a layman to assist him, with promise of great reward in the event of success. Complete success was obtained after a visitation, accompanied by the sight of a cloven hoof, and much smell of sulphur. So the method of sulphuring would not appear to have a very reputable origin!

H. E. DURHAM

half-packet should be added. The quantity in the packet weighed 242 grams, which when added to 100 gallons would give the proportion of 53.26 grams per hectolitre, whilst the extra dose would raise it to 79.89 grams per hectolitre. The proportions of SO₂ would then be 212.5 and 318.7 milligrams per litre respectively. These figures may be compared to the present French legal limits—10 grams of alkaline bisulphite per hectolitre and 100 milligrams of total SO₂ per litre.

It is probably not always easy to determine the form in which an addition in the form of sulphite of the alkalis has been made. In the case of the calcium salt (e.g. the above mentioned preservative) there should be a recognisable increase in the calcium when a considerable dose has been used; thus with the dose of 53.26 grams per hectolitre which has been considered in relation to the amount of SO_2 , there would be an increase of about 13.6 grams of calcium per hectolitre or about 13.6 milligrams per 100 c.c. In the case of sample No. 18 (vide table, p. 29), which has evidently been heavily sulphited, there was an unusual amount of calcium, viz. 28 mgrms per 100 c.c.; this is three to four times the amount I have hitherto found in ciders or in apple musts, and indeed surpasses the quantity I found, 25.7 mgrms, in the must of the "Holmer" pear. I may add that the beverage in question is advertised as having been made from the apple, although its aroma somewhat suggests that pears have been used in its manufacture. The extra dose of the sulphite preparation (vide above, 79.89 grams) would account for about 20 mgrms of Ca per 100 c.c., and this would leave about 8 mgrms for a normal amount of apples and many sorts of pears.

Where the salt used is metabisulphite of potassium, great difficulty would be experienced in determining the source of the SO_2 ; this is seen when it is considered that the French allowance of 10 grams per hectolitre only means an increase of about 3.5 milligrams of potassium per 100 c.c. (or the 1907 limit of 20 grams = about 7 mgrms of potassium), both of which fall within the limits given by Kulisch as normal¹. However, when large doses of the salt have been employed, such as to give an SO_2 proportion of 100 mgrms per litre, there would be an increase of about 60.6 mgrms of potassium, and so on for larger amounts.

Only five of the samples under review have been examined for the determination of the calcium and magnesium, and none for that of

¹ Kulisch gives (Landwirthschaftliche Jahrbücher, xIX. p. 102):

 Apple cider Calcium
 0.0056—0.0132 g. per 100 c.c.

 ,,
 Potassium
 0.133 — 0.182 ,,

potassium; from these analyses it appears that probably the sulphurous acid was introduced as such or as the potash salt.

Excessive sulphates. An excessive proportion of sulphates may indicate a sulphited product which has become oxidised from air exposure. Examples of this are afforded by some analyses of the remains of bottles which had been opened for other analyses and then recorked and kept.

Specimen A when obtained in 1907 was tested by the fermentation test and then showed the abnormal flocculent growth of yeast without fermentation; for the sugar titration showed $3^{\circ}/_{\circ}$ both originally and again 40 days later; eventually fermentation set in and the liquor went "dry." Another bottle of the same, kept for a year, and which had been opened for sometime but recorked with an ullage, gave an amount of sulphate equivalent to 201.1 mgrms of SO₃ per litre, about half of which probably proceeded from an oxidation of added SO₂. It was a fancy-named beverage and was thought to have been treated with formaldehyde as well.

Specimen B was the remains of a bottle which had become acetous and gave as sulphates 176.4 mgrms per litre SO₃; probably there were about 68 mgrms of SO₂ originally.

These figures may be compared with those of Nos. 18 and 21 in the table (p. 29), in both of which the sulphate content is excessive.

At the other end of the scale are the samples Nos 1, 2, 7, and 22 in which the sulphates are extraordinarily low; it may be noted that these all come from the same county and consequently the peculiarity is probably due to soil and variety of apple.

Analytical details of the samples examined.

In some of the samples which were examined it was known that no sulphuring whatever had been used. In all of these cases a minute amount of barium sulphate, far too small in amount to weigh, was found in the treated distillate. As the iodine solution by itself, in much greater quantity than that used in these distillations, gave no trace of precipitate with baric chloride, it may be supposed that a minute amount of some organic sulphur compound may be present and volatilised with the vapours in distillation and subsequently oxidised by the iodine.

In the subjoined table all the quantities are given as milligrams per litre. This list would have been considerably extended, but unfortunately many samples were tested with an iodine solution which subsequently was found to contain sulphate from an impure preparation of potassium iodide; since the amount of iodine solution used had not been measured in each case these had to be rejected, although they showed that the samples were highly sulphited, both by the smallness of the error due to impurity and from the decolourisation of the iodine solution.

I. Samples of known sulphuring-free history.

		burbher of mouth surburned noo m	
Sample No.		Sulphurous acid (SO ₂)	Sulphuric acid (SO ₃)
1	Cider	? minute trace	30.86
2	,,	,,	10.26
3	,,	,,	84.36
4	,,	,,	73.29
5	,,	,,	113.20
6	Avera	ge according to Wagner (German cide	rs) 90·00
II. Samples from open market containing SO_2 .			
7	Cider	10.62	17.15
8	,,	42.00	60.00
9	,,	222.00	68·00
1 0	Perry	51.30	51.34
11	Cider	194.0	92.3
12	,,	123.2	9 9·9
13	Perry	97•5	80.4
14	Cider	56.2	61.7
15	,,	211.4	58.2
16	,,	208.2	64.2
17	,,	181-2	61.7
18	Fancy	y name 242·4	198.0
19	Cider	599.0	87.4
20	"	68.2	56.1
III. Samples of unknown origin (from bottlers).			
21	Cider	? trace	137.8
22	,,	33	25.7
23	,,	,,	106.3

It will be obvious that there is a considerable latitude of idea amongst cider-makers as to the amount of sulphurous acid needed to produce "pure" cider.

The question may well be asked why some ciders (Nos. 1, 2, 3, 4, and 5) can be prepared without sulphites, and that in the absence of salicylic acid or other added preservative, whilst others contain nearly 600 milligrams to the litre. It may be inferred on the one hand that the addition of preservatives is not necessary, and on the other, that negligence or want of control in the extent to which they are added should be regulated. Moreover, many of the sulphited samples are guaranteed to be free from all additions to the apple juice, whence they were eventually derived ; such statements should not be permissible.

We may now turn seriatim to the various samples which deserve special mention. No. 7, a "pure" product, is evidently free from deliberate additions of sulphite; it was also free from salicylic acid. Its "purity" apparently included an addition of some form of oak tannin, for it did not give the normal greenish reaction with ferric chloride; when precipitated with lead acetate (which carries down gallo-tannic acid) the filtrate gave the normal greenish reaction of apple tannin.

Nos. 8, 9, 10 were from the same maker and also free from adulterations. The differences in amount of SO_2 are partly explained by the fact that No. 9 was more sugary than the others, and with regard to the perry I am informed that this maker has the reputation of buying his perry ready made.

Nos. 11, 12 and 13 are the products of one maker, who though working in the same region as the maker of No. 14, uses twice to thrice as much SO_2 .

Nos. 15 to 18 are all products of one firm, which is evidently addicted to the use of the acid in considerable proportions. Further remarks upon No. 18 have already been given, but the high values of both sulphurous and sulphuric acids may again be pointed out.

No. 19 stands out from all the other samples by its huge amount of sulphite. This is the kind of preparation which may help to cause consumers to consider that cider is a beverage which causes gastric discomfort. Several estimations were made from different bottles to confirm the high figure, e.g. Sample "B," p. 22.

No. 21 is an example of an absolutely objectionable concoction, for though no sulphur as sulphite was found, nor indeed salicylic acid, its sweetness was found to depend upon the presence of saccharin. The high figure of the sulphuric acid suggests that originally the sample must may have been sulphited and subsequently oxidised.

No. 22 when tested by ether shake was found to be free from salicylic acid, but the residue gave an unusual dirty greenish precipitate with ferric chloride, of unknown nature.

No. 23 was not tested for antiseptics other than SO_2 .

Suggestions and recommendations.

The suggestions and recommendations which follow may be divided conveniently into two groups; namely those which affect the addition

H. E. DURHAM

of preservatives generally to food products, etc., and those which affect the addition of the special preservative which forms the subject of this paper.

A. General regulation of preservatives in food products.

1. Further and more precise regulation is needed in this country at an early date.

2. The presence of added preservatives in food products should only be permitted with declaration of the nature of the preservative. Exceptions of defined substances, such as common salt, should be made.

In declarations such phrases as harmless or unobjectionable additions should not be permitted.

3. Where quantitative distinctions are made, the official method of estimating the quantities should be defined.

4. Where prosecutions are undertaken for illicit additions to food products, not only the vendor and maker of the food product, but also the vendor, maker or importer of the chemical in question should be proceeded against¹.

5. Manufacturers or importers of food products which contain permissible amounts of permissible chemicals should be required to take out licenses to allow them to use these chemicals, and their works or premises should be registered and liable to visits of inspection by the health authority.

6. Manufacturers or importers of permitted chemicals should also be required to license and register themselves. Further when supplying non-permitted agents, which are capable of use as additions to food products, they should obtain information as to the use to which the said substances are destined.

7. Firms or individuals who trade by supplying analyses or certificates of purity should be compelled to report the presence of added permissible or non-permissible preservatives. And they should be liable to prosecution for neglect or misrepresentation.

B. The regulation of sulphurous acid and sulphite preservatives in cider and perry.

1. The addition of sulphurous acid and sulphites to cider and perry needs regulation.

¹ Vide Durham, Journ. Roy. Inst. Pub. Health, xvi. 1908, p. 293.

2. The permissible limit of addition of the substances should be expressed as "total sulphur dioxide" obtained by distillation with phosphoric acid.

3. Judging from the practice of some makers, the addition of sulphites or other preservative is unnecessary, and from that of other makers whose products only contain relatively small proportions, the maximum legal limit of "total sulphur dioxide" should be low; and at any rate not exceed 100 milligrams per litre.

4. Ciders and perrys containing more than a "trace" of sulphite should be labelled with a declaration to that effect. For practical purposes the word "trace" might be defined as "less than 10 (or possibly 20) milligrams of sulphur dioxide per litre."

5. Such terms as "pure," "free from preservatives" and the like should not be permitted for ciders which contain more than a "trace" of sulphur dioxide.

6. Cider-makers who desire to use sulphite or other preservatives should be required to take out license and registration.

7. Cider-makers and vendors who are unlicensed and in whose products more than a "trace" of sulphur dioxide is found, should be liable to prosecution.

8. Cider-makers or vendors, who are licensed for sulphite additions, but in whose products more than the maximum permitted limit of sulphur dioxide is found should be liable to prosecution.