INSTABILITY IN POTABLE SPIRITS. II.*—Rum and Brandy

By L. A. WARWICKER†

Instability in rum and brandy is partly inherent, partly caused by contamination during maturation, and partly due to bad filtration techniques. Instability occurs as deposition with metallic contamination and alteration of the pH to critical values. It is distinct from the formation of inks. Chill-proofing is not sufficient in itself to provide a stable product if sufficient contamination occurs afterwards. Ion-exchange in the H^+ form removes an instability factor. The presence of amino-acids in the hydrolysis products of the deposit, but not in the unhydrolysed deposit, suggests that it contains protein.

Introduction

Manufacturers of potable spirits have found difficulty in obtaining a ' bright ' product, or one that will retain its brightness for several months. A typical instability is ' greying ' (the development of cloudiness or haze), which is produced when the spirit is subjected to low temperatures. The process of haze production is reversible since, when the temperature of the spirit is raised, the haze will disappear, providing that no deposition has occurred. If the spirit has developed a deposit it is said to be ' thick ' and complete reversal is very unlikely.

The purpose of these investigations was to determine the causes of each instability and to suggest means by which the stability of spirits might be improved.

Instability in a spirit could be an inherent quality of the individual spirit concerned, it could be caused by contamination during manufacture or it could be developed during maturation.

Rum, obtained from the fermentation and distillation of sugar cane, and brandy, obtained from the distillation of wine, usually grape wine, are both products of distillation from a pot still.

Three rums were investigated, Brand D (Demerara), Brand J (heavy Jamaican) and Brand S (Trinidadian), all being blends of ' straight ' rums from the respective areas. The brandy (Brand G) was French of ordinary quality, which had been chill-proofed at approximately proof strength. Brands D and S were of natural colour, while brands J and G were coloured to a standard.

The production of rum and brandy has been described by a number of authors.[1–4]

Experimental

(A) *Analysis of the rums and brandy*

Gallon samples of each brand were filtered through membrane filters to ensure a bright filtrate. Samples of each were analysed for alcohol, potassium, sodium, tannin, fusel oil, colour, calcium, magnesium, iron, copper, phosphorus, total acidity, pH and total cationic species by methods previously described.[5] They were also analysed for esters and aldehydes.[6]

(B) *Addition experiments*

Samples were inspected daily for the first 4 weeks, then weekly for the next 2 months, and then monthly.

(a) *Cationic and anionic additions.*—To 50-ml. samples of each brand were added various salts (see Table II) in amounts to increase the cation concentration by 5 or 10 p.p.m., or to increase the anion concentrations by 10 p.p.m.

(b) *Addition of alcoholic and aqueous wood extracts.*—Extracts of Limassol oak and wood used for rum maturation were prepared as previously described.[5] After concentration and filtration, the filtrates were analysed for tannin, iron, copper, calcium, magnesium, phosphate and pH. The residual wood in the extraction thimbles was air-dried and weighed to give an approximate loss of weight.

To a 50-ml. sample of each brand were added 3–4 drops of the appropriate alcoholic (or aqueous) wood extract. In a control experiment water was used in place of extract.

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† Present address: Nabisco Foods Ltd., Welwyn Garden City, Herts.

(c) *Additions to 'doubly cation-exchanged' rums and brandy.*—Samples of the 'doubly cation-exchanged' brands were prepared by ion-exchange and additions made as previously described.[5] A further experiment was carried out by adding sufficient of a 200 p.p.m. (cation) solution of calcium acetate, copper acetate or zinc acetate to increase the cation content by 10 p.p.m.

(d) *Addition to 'chilled' rums and brandy.*—All the brands at 70° proof were kept for 3 days at −15° and filtered cold. To 50-ml. samples of the filtrates were added 2·5 ml. of an aqueous solution of magnesium acetate (200 p.p.m. Mg) to increase the magnesium content by 10 p.p.m. Control experiments were set up with 2·5 ml. of water.

(C) *Alteration of pH*

The pH of 50-ml. samples of each brand were adjusted to definite values between 4·9 and 10·6 by the use of N- and 0·1N-sodium hydroxide and 10% aq. ammonia. The pH of each sample was checked by pH meter and the samples inspected daily for 1 month and then weekly.

(D) *Contact with metals*

To 50-ml. samples of each were added pieces of metal approximately 1 sq. cm. surface area (aluminium, brass, copper, iron, lead or zinc). The samples were inspected daily for 1 week, then weekly for 1 month and finally monthly up to 4 months. Fusel-oil-free alcohol and the metals were used as controls.

(E) *Analysis of deposit*

(i) The sludge from the bottom of a rum vat, after reduction to bottling strength, was drawn off and allowed to settle and the supernatant rum decanted as far as possible. The residue was membrane-filtered and washed several times with distilled water. This small quantity of deposit was subjected to routine qualitative inorganic and organic analysis, after extraction with ether and removal of the latter by vacuum distillation. A sample of the deposit was hydrolysed with 6N-hydrochloric acid in a sealed-tube for 4 h. in an autoclave at 15 p.s.i. The filtrate was diluted with water and concentrated under vacuum to remove as much excess acid as possible. This was repeated several times. The pH of the concentrate was then adjusted to 5·0 with aq. ammonia, some ferric hydroxide which precipitated was removed by centrifuging. The concentrate was spotted with an Agla micrometer syringe on to Whatman No. 1 chromatography paper and run by ascending chromatography in two solvents. The first solvent was a 60 : 15 : 25 mixture of n-butanol, glacial acetic acid and water (upper phase of Partridge solvent) and the second a saturated mixture of phenol in water. After being air-dried, they were developed by spraying with 0·2% ninhydrin in acetone, to detect amino-acids.

(ii) A sample of the deposit was dissolved in hot glacial acetic acid, filtered and the solution concentrated by vacuum. A portion of the concentrate was chromatographed for amino-acids as previously described. A sample of the white solid formed in the acetic acid solution was filtered off and divided in two. Half was refluxed for ½ h. in 50% aq. sodium hydroxide and the other in alcoholic potassium hydroxide. The products of hydrolysis were then chromatographed with the butanol solvent and examined for tannins by ultra-violet light and developed with ferric ammonium sulphate solution.

Results

The analyses of the various brands are detailed in Table I and the effect of cationic additions to the spirits is shown in Table II. Anionic additions did not give any deposit, in most cases, up to 1 year. The results for pH values are in the order Brand D, J, S and G, respectively (here and below pH values are given in brackets) : sodium acetate (4·50, 3·50, 4·62, 4·05) ; sodium chloride (4·33, 3·52, 4·55, 3·60) ; potassium dihydrogen phosphate (4·28, 3·28, 4·60, 4·07) ; sodium nitrate (4·40, 3·50, 4·63, 4·00) ; disodium hydrogen phosphate (4·50, 4·14, 4·75, 4·10) ; sodium sulphate (4·29, 3·62, 4·50, 3·98). With sodium carbonate, brand J (3·65) gave a deposit after 2 months and brand S (4·78) after 5 days. Brands D (4·90) and G (4·15) did not give a deposit up to 1 year.

Table I

Analysis of spirits

	Brand D	Brand J	Brand S	Brand G
Real strength, ° proof	70·0°	69·8°	70·0°	70·0°
,, ,, , % volume 20° C	40·08	39·97	40·08	40·08
Apparent strength	70·0°	68·9°	70·0°	67·5°
Obscuration	nil	0·9	nil	2·5
Colour units	0·86	12·95	4·24	13·25
Tannin, p.p.m.	44	225	170	425
Sodium, p.p.m.	3·7	24·5	6·6	24·5
Potassium, p.p.m.	2·2	13·5	22·5	19·5
Calcium, p.p.m.	3·5	10·0	0·8	13·5
Magnesium, p.p.m.	—	—	1·6	—
Iron, p.p.m.	0·7	1·3	0·3	0·6
Copper, p.p.m.	0·1	0·9	0·9	1·0
Phosphate, p.p.m.	nil	nil	nil	nil
pH	4·30	4·13	4·38	4·41
Total acidity × 10^{-3}N	0·95	5·9	3·5	3·5
Total cationic species × 10^{-3}N	0·1	0·7	0·95	1·05
Fusel oil, g/100 l. of absolute alcohol	125·3	241·2	258·0	205·4
Esters, ,, ,,	7·9	115·8	44·1	81·1
Aldehydes, ,, ,,	9·0	74·4	23·6	62·1

The analysis of the wood (alcoholic and aqueous) extracts is shown in Table III. When these were added to the spirits no typical deposit was observed up to 6 months. The samples with added aqueous wood extract were bright except for S. pH values after aqueous and alcoholic additions, respectively, were brand D (4·65, 4·43); brand J (3·75, 3·98); brand S (4·35, 4·48); brand G (3·91, 4·88).

The addition of Mg^{2+}, Ca^{2+}, Cu^{2+} and Zn^{2+} as acetates to the double H^+-exchanged brands did not produce a deposit in any of them after 6 months. The pH values ranged from 3·15 to 3·82. Alteration of the pH of the doubly H^+-exchanged brands to approximately 5·0 with

Table II

Observations on deposition after cationic additions

Ion added	Ion increase, p.p.m.	Brand D		Brand J		Brand S		Brand G	
		pH	Observation	pH	Observation	pH	Observation	pH	Observation
Aluminium (as sulphate)	5	3·87	Yellow 1 day	3·82	2 months	3·75	Brown 1 day	4·11	No deposit
,, ,,	10	—	—	3·62	3 weeks	3·66		—	1 year
Calcium (as acetate)	5	4·62	1 month	3·89	3 weeks	4·32	5 days	4·26	,, ,,
,, ,,	10	4·90	5 days	3·00		4·51	1 day	4·60	
Copper(II) ,,	5	4·40	No deposit 1 year	3·76	Heavy brown in 3 days	4·24	No deposit 1 year	4·50	,, ,,
,, ,,	10	4·48		3·95		4·22	Heavy brown 12 days*		
,, (as sulphate)	5	4·1	Small 10 months		—	—	—	4·20	
Iron(III) (as chloride)	5	3·75	Black and darkening 5 days		—	—	—		Darkening but no deposit 1 year
,, ,,	10	3·59			—	—	—	3·90	
Lithium ,,	5	4·36	No deposit 1 year		—	—	—	4·21	No deposit 1 year
,, ,,	10	4·30			—	—	—	4·10	
Magnesium (as acetate)	5	4·95	5 days	4·15	7 days	4·41	5 days	4·15	12 days
,, ,,	10	5·18		4·30	Heavy 3 days	4·53	1 day	4·41	5 days
Tin(II) (as chloride)	5	2·60	Pale 1 day	—	Granular 10 months	3·99	No deposit 1 year	3·76	Slight 2 months
,, ,,	10	2·30		2·58		4·05		3·63	Brown 1 day
Zinc (as acetate)	5	4·58	Slight 2 months	3·97	3 weeks	3·95	No deposit 1 year	4·30	No deposit 1 year
,, ,,	10	4·80	Slight 5 days	3·97		4·08		4·36	2 months

* 33 p.p.m. Cu added in error

Table III

Analysis of wood extracts
(results as p.p.m.)

	Iron	Copper	Magnesium	Calcium	Tannin	Phosphate (PO_4^{3-})	Loss %	pH
Rum wood, alcohol extract	0·2	1·2	3·0	12·5	30,375	7·6	3·9	3·85
,, ,, water ,,	0·2	0·7	41·6	30·9	14,375	12·3	6·1	3·30
Limassol oak, alcohol extract	0·2	0·4	13·1	28·5	58,750	42·0	5·8	3·62
,, ,, water ,,	0·1	0·8	4·4	6·2	33,750	38·3	12·3	3·20

sodium hydroxide and aq. ammonia respectively did not develop a deposit in brand D (5·00, 8·91) ; brand S (5·28, 6·06) and brand G (4·93, 5·0). Brand J developed a deposit in 3 days (4·95) with sodium hydroxide and 2 weeks (5·12) with aq. ammonia. Addition of ethanolic wood extract did not produce a deposit with brand S (4·79) or G (3·85) after 6 months. Brand D developed a deposit in 3 days (4·91) and brand J in 3 months (4·92).

The addition of magnesium ions to the chilled spirits produced a deposit in 2 weeks in brand D (5·19) and 1 week in brand S (4·35). No deposit was formed in brands J (3·98) or G (4·21) after 6 months.

The alteration of the pH, by sodium hydroxide, of brand D to 5·10, 6·25 and 7·40 resulted in a granular deposit which after 9 months became typical and fluffy and remained the same up to 1 year. The samples at pH 9·70 and 10·55 remained granular, and at pH above 7·0 the colour darkened. Brand J at pH 5·10 developed a deposit in 1 day, but at pH 6·05 and 10·40 only a slight granular deposit was found after 1 year. From pH 6·10 the colour began to darken slightly. Brand G (4·90) developed a deposit after 3 weeks, while at pH 6·10, 7·45, 9·65 and 10·70 it had only a slight granular deposit after 1 year. The colour increased as the pH increased above 6·10. Brand S at pH 5·0 and 6·1 developed a deposit in 1 and 4 days respectively, but at 10·5 only a slight granular deposit was found after 1 year. The colour darkened above pH 6·1 and it became intense at pH 10·5.

The effect of metals in contact with the spirits is shown in Table IV. All the samples of brand J became practically colourless. Similarly with sample G the samples in contact with aluminium, lead and zinc were practically colourless. In the control experiment only aluminium (8·00) and zinc (9·22) showed a slight deposit after 3 months. The other pH values were for brass 8·73, copper 9·22, iron 8·95 and lead 9·26.

The deposit did not contain any significant quantities of metals. Paper chromatography

Table IV

Effect of metals on spirits

	Brand D	Brand J	Brand S	Brand G
Aluminium	Very slight granular deposit 4 days (4·93)*	Slight deposit 1 day Heavy deposit 4 months (4·69)	Slight deposit 1 day Heavy deposit 1 month (4·71)	Slight deposit 1 day Deposit 4 days (5·11)
Brass	Slight granular deposit 4 months (5·62)	Heavy deposit 4 months (5·01)	Slight deposit 1 month Deposit 4 months (4·78)	Very slight deposit 4 months (4·40)
Copper	Deposit 4 months (5·55)	Haze 1 month Heavy deposit 4 months (5·37)	Slight deposit 1 month Heavy deposit 4 months (4·86)	Very slight deposit 4 months (3·95)
Lead	Very slight deposit 4 days Heavy deposit 1 month (5·80)	Heavy deposit 1 day (5·73)	Heavy deposit 1 day (5·31)	Heavy deposit 1 day (4·92)
Zinc	Deposit 1 month (6·60)	Slight deposit 1 day Heavy deposit 1 month (6·83)	Slight deposit 1 day Heavy deposit 1 month (6·53)	Heavy deposit 1 day (6·45)

* Figures in brackets are pH values

of the deposit dissolved in acetic acid showed that it did not contain any amino-acids. Tests for polyphenols with ultra-violet light, diazosulphanilic acid and ferric ammonium sulphate produced negative results. The acid hydrolysis of the deposit gave three amino-acids (literature R_F values in parentheses), which were confirmed by running a number of standard amino-acids.

	R_{F_1}	R_{F_2}
L-Serine	0·22 (0·22)	0·34 (0·35)
Proline	0·34 (0·34)	0·88 (0·90)
Phenylalanine	0·60 (0·60)	0·84 (0·84)

The deposit after aqueous or alcoholic alkaline hydrolysis and then chromatography did not show the presence of any tannins with ultra-violet light or ferric ammonium sulphate.

Chromatography of the wood extracts did not show the presence of any amino-acids, but only a large number of tannins which were not clearly separated.

Two brands of unchilled brandy and a brand-S rum were treated by the Petrosyan technique,[7] when a trace of DL-leucine was found in each.

Discussion

The present work was undertaken after observations on brandy which, shortly after a clarifying filtration, ' threw ' a deposit which was heavier than that in the unfiltered spirit. The occurrence of these deposits was related to the type of filtration medium used. The increase in pH after filtration indicates a replacement or removal of an acidic part of the spirit. Filter-pads acid-washed by the manufacturers to remove as much extractable magnesium as possible are now used. Calcium pick-up in wine from filter-pads and filter-aids is mentioned by De Soto & Warkentin.[8] Previous work[5] indicates that a magnesium pick-up contributes to instability, but instability is not caused by ' inks ' formed from excess iron and the tannin extracted from casks during maturation. It was shown that when the iron–tannin product ratio was increased to nearly seven times the normal value darkening occurred, but there was no deposition.

Aluminium salts produced a deposit with pure aqueous alcohol, probably due to hydration of the Al^{3+}. Addition of Fe^{3+} to the four brands resulted in blackening, which is characteristic of iron contamination. The anionic addition of carbonate produced a deposit in brands J and S. Brand D (pH 4·8) to which calcium and zinc ions were added showed typical deposition after a few days, but adjustments of the pH to 5·1 with sodium hydroxide and 4·9 with carbonate did not lead to typical deposit formation within 9 months. After 11 months the deposit in brand D with pH adjusted with sodium hydroxide assumed a typical form. In brand J, addition of calcium, magnesium and zinc ions (pH 4·0) and adjustment of pH to 5·1 with sodium hydroxide resulted in typical deposit formation, but this result may be influenced by the initial high calcium content in brand J. Brand S showed typical deposition as a result of additions of calcium and magnesium (pH 4·0) and pH adjustment with sodium hydroxide to 4·9 : addition of copper ions caused deposition, but the amount (33 p.p.m.) added was due to a dosing error. Brand G also threw typical deposits after magnesium and zinc additions (pH 4·4) and after pH adjustment with sodium hydroxide to 4·9.

The various cationic additions to the doubly H^+-exchanged brands did not induce deposition. The adjustment of pH (4·95 and 5·12 with sodium hydroxide and aq. ammonia respectively) only induced deposition in brand J. The addition of alcoholic wood extract and adjustment of pH with sodium hydroxide to 5·0 resulted in a deposit in brands D (4·91) and J (4·92). Since the pH values after cationic additions were not close to the critical value (4·9), no deposition would be expected. Adjustment of pH to the critical values only resulted in one brand throwing a deposit and it can be concluded that the remaining brands have had an instability factor removed by H^+-exchange. Since brand D shows deposition after addition of wood extract and pH adjustment, an instability factor must be reintroduced in the extract. Brands S and G did not throw a deposit and therefore this factor cannot have been introduced in sufficient quantity to cause instability.

On chilling and subsequently adding magnesium, a deposit was formed in brands D (pH

5·19) and S (4·35) but not in J (3·98) or G (4·21). The deposit in brand D took 1 month to form, whereas the unchilled material deposited in 5 days (5·19). Chilled brand S threw a deposit in 1 week and the unchilled in 1 day (4·53). Unchilled brands J and G gave a deposit in 3 days (4·30) and 5 days (4·21) respectively. Brands D and S have been partially stabilised by chilling, but brands J and G have been protected by it against the effect of subsequently added instability factors. The quantity of deposit from the chilled was much less than from the unchilled samples.

The action of all the metals on rums resulted in a considerable loss of acidity. Brand G had a loss of acidity except with copper and brass. In most cases the spirit became practically colourless, as though a fining action had taken place. The dissolution of metal increased the pH above the critical value and hence deposition occurred. Walter[9] showed that corrosion of metals by alcoholic liquids depends on the acid content, the type of metal, the period of contact and the temperature. He also indicated that alcoholic liquids (40–50% by volume of alcohol) attack iron more easily and zinc, copper, aluminium, tin and bronze less easily. The corrosive effect of rum and brandy on aluminium has been discussed.[10, 11] Adzhemyan[12] states that an acid content of 0·1–0·2 g./l. in brandy will dissolve such metals as copper, aluminium, zinc, cast-iron and brass, the last two being most soluble, reaching 4–8 p.p.m. in brandy. Rankine[13] confirms that the extent of copper contamination is closely related to the total acidity: he gives an example of brandy left in a brass pump for 24 h. which showed an increase of 150 p.p.m. of copper. He states that a copper–tannin combination forms during maturation and flocculates as a brown deposit. This reaction depends on pH and is more complete at pH 4·2, less between 4·2 and 2·5, and does not occur at pH below 2·5. He also shows that the copper is cationic in young brandy and cationic and anionic in matured.

The extraction of the woods was more severe than would occur during maturation. The analytical results show that the alcoholic extracts contain nearly twice as much tannins as the aqueous. The latter has a fairly high calcium and magnesium content. Only the alcoholic extracts produced a deposit and these therefore contain a factor which causes or aids instability. These extracts contain water-insoluble components which come out of solution on breaking down. Support is given by observations with American whisky[14] that the higher the barrelling proof, the greater is the tendency to show higher turbidities when reduced to bottling strength. During maturation the alcohol content falls as alcohol molecules diffuse through the wood by capillary attraction. During this time the total solids content increases indicating that extraction has taken place.[15, 16] Various authors[17–19] have discussed the effects of ageing on the alcohol, the oak-wood and the class of compounds extracted and formed during this process.

Neither the wood extracts nor the deposits gave a positive result for steroids by the Liebermann–Burchard, Salkowsky–Hesse or Tortelli–Jaffe tests, so the presence of a steroid, as found in American whisky,[20] is unlikely. Since only the hydrolysed deposit contained amino-acids and not the unhydrolysed deposit or wood extracts, the deposit is possibly of a proteinaceous nature. This is further supported by the critical pH having values in the range of the isoelectric point of proteins.

The use of various finings did not improve the stability of the spirits and in a number of cases it deteriorated. A chilling process appears to be the best method of stabilising rums and brandies and hydrogen-ion exchange can be used for special treatment.

Conclusions

From the experimental work concerning the nature of the deposits in rum and brandy, it is concluded that the production of 'inks' is not a cause of deposition, but extraction of magnesium salts from filter-pads and filter-aids contributes to the formation of a deposit. From the finding of amino-acids after hydrolysis it is concluded that the deposit must be at least partly protein and this is further supported by the critical pH values for deposition being 4–5, which are near the isoelectric points for proteins. There are many tannins extracted during maturation and the deposit may contain non-hydrolysable ones. Hydrogen-ion exchange removes a factor causing instability, and both this and chilling stabilise rums and brandies. Chilling must be at final strength otherwise there is not sufficient removal of water-insoluble matter to avoid deposition, if the spirit is further reduced.

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Research Dept.
W. & A. Gilbey Ltd.
Oval Road
Regent's Park
London, N.W.1

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INSTABILITY IN POTABLE SPIRITS. III.*—Gin and Vodka

By L. A. WARWICKER†

For good quality gin and vodka a 'clean' spirit is essential. Prolonged contact with a number of metals, or contamination with metallic salts, is to be avoided. There is no critical pH for the formation of a deposit as in other potable spirits. The deposit formed in vodka is calcium carbonate, but deposits of calcium and magnesium sulphate and magnesium carbonate are also possible. The solubility of calcium carbonate in different concentrations of alcohol is compared with values for Russian vodka and the effect of large concentrations of other ions is considered. The use of demineralised water is recommended for both gin and vodka. Where this is not possible, careful monitoring of softened water is essential.

Introduction

Gin and vodka are potable spirits which do not require maturing before being consumed. Some Polish vodka is left in Tokay casks and one London gin is left in wood to mature. Both

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* Part II: preceding paper
† Present address: Nabisco Foods Ltd., Welwyn Garden City, Herts.