

54/04      May 2004

## **SURVEY OF SULPHUR DIOXIDE IN SOFT DRINKS**

### **Summary**

- A survey of sulphur dioxide and sulphites (E220-E228) in soft drinks has been conducted to ensure manufacturers are complying with legislation.
- The samples consisted of concentrates based on fruit juice (squashes), lemon and lime juice, and concentrates based on fruit juice containing not less than 2.5 per cent barley (barley waters). The levels of sulphur dioxide in all the tested samples were within the statutory limits. This indicates that all the manufacturers of soft drinks included in this survey are complying with this particular aspect of food additives legislation.
- A further survey of sulphur dioxide in sausages and burgers is currently being carried out by the Agency. The results of this survey are due to be published later this year.
- As sulphites are chemically unstable in foods and are often lost as sulphur dioxide gas, further investigation into the fate and stability of sulphites in foods is due to be carried out by the Agency. The findings of that research will be used with the results of this survey to further refine intake estimates for sulphites, and these will be used to inform future EU discussions on maximum permitted levels of sulphites in foods.

## **Background**

Sulphur dioxide is added to food in the form of sulphites (E220-228) and is a widely used food preservative. The main function of sulphur dioxide and sulphites is the control of microbial spoilage.

The use of sulphites in foods is controlled by The Miscellaneous Food Additives Regulations 1995 (Statutory Instrument (SI) 3187)<sup>1</sup> as amended. Sulphites are permitted in certain soft drinks, and in breakfast sausages and burger meat with a minimum vegetable and/or cereal content of 4 per cent, as well as in other foodstuffs. The maximum permitted levels for sulphites are expressed as sulphur dioxide, and the maximum level in soft drinks is 350 mg/l for lime and lemon juice and concentrates based on fruit juice containing not less than 2.5 per cent barley (barley water), and 250 mg/l for other concentrates based on fruit juice or comminuted fruit.

Soft drinks, sausages and burgers are likely to be the major contributing factor to sulphur dioxide intakes, due to the levels of consumption of these foods and the levels of sulphites permitted in them. This survey is looking at the levels of sulphur dioxide in certain soft drinks and a second part of this survey is examining the use of sulphites in breakfast sausages and burger meat and is due to be published later this year.

Sulphur dioxide and several forms of sulphites, which generate sulphur dioxide when used in food, have been considered in this survey. These include sulphur dioxide (E220), potassium bisulphite (potassium hydrogen sulphite) (E228), potassium metabisulphite (E224), sodium bisulphite (sodium hydrogen sulphite) (E222), sodium metabisulphite (E223), sodium sulphite (E221), calcium sulphite (E226) and calcium hydrogen sulphite (E227). As all the above can convert to sulphur dioxide, sulphites are measured and expressed as sulphur dioxide.

Sulphites are normal products of the human body's metabolism and so when ingested the body is able to metabolise them efficiently. Animal studies have shown that very high levels of sulphites can lead to gastric reactions. As a result, in 1994 the European Commission's Scientific Committee on Food (SCF) set an acceptable daily intake (ADI) for sulphur dioxide of 0.7 mg/kg body weight/day<sup>2</sup>. The ADI is the amount of food additive,

expressed on a body weight basis, that can be ingested daily over a lifetime without appreciable health risk.

There is the potential for sulphur dioxide to induce bronchoconstriction or bronchospasm in people prone to asthma, and this may occur at low levels in very sensitive individuals, but they are already likely to avoid foods that contain sulphites. Under food labelling law, when sulphites are used in a product they must be declared on the label either by name or e-number. New EU requirements regarding the labelling of allergens in foodstuffs will come into force in the UK on 25 November 2004. These will extend the requirement for ingredient listing for prepacked foods, including alcoholic drinks, to include certain substances identified as potential allergens. The new Directive requires the presence of sulphur dioxide and sulphites or their derived ingredients to be indicated on labels by specific reference to sulphur dioxide and sulphites when present at or above 10mg/kg or litre.

## **Methodology**

The determination of sulphites and sulphur dioxide in a food sample can be difficult as sulphites and sulphur dioxide are volatile compounds. Many methods exist for the measurement of free, total, and combined sulphites. The main methods used for their determination are based on the Monier-Williams type procedure with either volumetric or gravimetric end-point determinations. This procedure is based upon distillation of sulphur dioxide from an acidic medium. Sulphur dioxide is then determined either by titration (volumetric method) or by weighting the barium precipitate, having added barium chloride.

## **Samples and Timing**

Ventress Technical Services (VTS) Ltd., purchased seventy-five samples of soft drinks between November 2003 and January 2004 from 5 regions in the UK. Analyses of all samples were completed by the end of January 2004. The results were collated, checked and reviewed in February and March 2004.

Samples were obtained from the following five locations in the UK:

- Belfast and district (10 samples),
- Cardiff and district (9 samples),
- Glasgow and district (10 samples),

- London (26 samples),
- Manchester and district (20 samples).

65 samples were purchased from supermarkets, 10 samples were purchased from independent retailers.

To allow sufficient time for analysis, products with less than six weeks to their best before end dates were not included in the survey. Of the 75 samples collected, 8 were lemon and lime juice, 2 were orange / lemon barley waters with more than 2.5 per cent barley and 65 were concentrates based on fruit juice (dilutable squashes). Each sample consisted of 3 bottles all bearing identical best before dates and batch codes.

All samples selected had to conform to the following criteria:

- Squash products were pre-packed dilutable soft drinks containing fruit juice, including high juice, barley water and cordials.
- Products had to declare at least one of the permitted preservatives E220-E228.
- Bottles of all sizes and packaging formats were included.
- Economy, standard, premium and own brand products of any fruit variety were included, as were no added sugar varieties. Local products, which were specific to a particular region, were sampled if identified to contain any of the preservatives listed above.
- The best before date and batch code for each sample had to be legible. Only undamaged, unopened, sealed bottles were purchased and there was no duplication of products.

Samples (Tables 1 & 2) were uniquely coded by region at the time of purchase:

- Belfast and district: samples with codes 211C001-211C010
- Cardiff and district: samples with codes 211C011-211C019
- Glasgow and district: samples with codes 211C021-211C030
- London and district: samples with codes, 211C031-211C055, and 211C020
- Manchester and district: samples with codes 211C056 –211C075

Following purchase, digital images of samples were taken and labelling and packaging details recorded. Any sample found to be in an unsatisfactory condition was removed from the survey at this time and not sent for analysis.

For eight samples, one bottle was inverted three times to ensure thorough mixing before being sub-divided into two containers. After removing all the air from the headspace, the caps were securely tightened and frozen. Each sub-sample was coded with the sample number and allocated a unique seal number. Once frozen to minus 18°C, one sub-sample was sent to Reading Scientific Services Ltd (RSSL) and the other to Durham Scientific Services (DSS) for analysis as external quality assurance. Any remaining product was left in the original bottle, the cap secured and the sample stored at minus 18°C to prevent loss of sulphur dioxide.

For the remaining 67 samples one bottle was sent, unopened to RSSL for analysis. The remaining two bottles were stored by VTS in a cool, dark location. Any sample remaining after analysis was frozen to prevent spoilage and loss of sulphur dioxide and stored at RSSL.

### **Standard Solutions**

A standard stock solution of approximately 1000 parts per million of sulphur dioxide was prepared by weighing (to two decimal places) 1.67g of di-sodium disulphite (sodium metabisulphite) into 100ml volumetric flask and made up to volume with water. This solution was standardised by iodine titration. 5 ml of the stock sulphur dioxide solution was pipetted into a 250 ml conical flask and 5 drops of 1 per cent starch indicator, or 0.2-0.3 g solid Iodine Indicator (BDH), added and dissolved. The contents of the flask were titrated with 0.05 Molar iodine to a blue-black end point. The titration was repeated until 2 consecutive titres within plus or minus 0.1 ml of each other were obtained. The mean of the titres was used. This stock solution was usable at the standardised strength for two days when stored at ambient temperature. The working solution (100 parts per million) was prepared on the day of use by pipetting 1 ml of the stock solution into 100 ml volumetric flask and made to volume with water.

### **Quality Control (QC) Samples**

An aliquot (30-40 g) of black pudding (the reference sample, free of sulphur dioxide, used for this method) was prepared. To this was added 0.5 ml of 1 per cent stock solution and this was analysed in the same way as survey samples. The SO<sub>2</sub> recovery obtained was

recorded and tabulated (using a reference sample analysis logsheet). The results were plotted on an appropriate Shewhart chart to assess the consistency of the results. Any results between 101.74 and 82.14 per cent were considered to be inside the control limits.

### **Sampling preparation**

Between 30-40 g of sample was used. No sample preparation was required before the analysis was carried out.

### **Distillation and Titration**

The samples were analysed by an in-house UKAS accredited method (TM195), which is based on the modified Monier-Williams method. The principle of the method is that sulphur dioxide is steam distilled from an aqueous acidic sample matrix using a distillation unit and trapped in an oxidising medium, to produce sulphuric acid. The sulphuric acid is titrated against aqueous sodium hydroxide. The steps to be followed in the procedure are as follows:

Between 30-40 g of sample was weighed directly into the reaction tube of the distillation unit. Using a measuring cylinder, 40 ml of neutralised hydrogen peroxide solution was measured into the collection vessel. 5 ml orthophosphoric acid was added from a measuring cylinder to the reaction tube, the tube was sealed and the distillation was started. When the distillation had finished the reaction tube was removed immediately and the tube was washed with water into the vessel. The contents of the collecting vessel were then quantitatively transferred into 250 ml conical flask using water to wash the vessel. The contents of the conical flask were titrated with 0.02 Molar sodium hydroxide, and Bromophenol blue indicator to a blue/green endpoint.

### **Quality Assurance**

- Prior to analysing any samples, a recovery check was performed on the system using liquid standards and Quality Control (QC) samples. If the recovery check gave a recovery of less than 90 per cent for liquid samples or between 101.74 and 82.14 per cent for QC samples it was investigated and rectified before any survey samples were analysed.

- The quality control (QC) samples and spiked samples were run with each analytical batch of samples to check system performance
- 10 per cent of samples were analysed in duplicate. All duplicate samples were considered acceptable as they were within the range expected once measurement uncertainty for this method was taken into account.

### **External Quality Assurance**

To ensure that results obtained by the laboratory carrying out the survey (RSSL) were of acceptable accuracy, a set of eight random sub-samples was sent to an independent laboratory (DSS) for 'blind' analysis. DSS used two methods of analysis, the method used for routine analysis is based on the 'Committee' distillation method and is similar to the one used by RSSL. Results from this were confirmed using gravimetric determination of sulphites by measuring precipitated barium sulphate. Details of both methods are available on request. Results corrected for recovery of the external Quality Assurance are reported for both methods in Table 3.

### **Confirmatory Analysis**

Samples that were found to contain levels of sulphur dioxide approaching or exceeding the maximum permitted level were also sent to DSS for independent confirmatory analysis (Table 4).

### **Analytical Uncertainty**

The measurement uncertainty, for sulphur dioxide at a specified level, was estimated for each laboratory. Both were asked to provide an estimate of its uncertainty level. The uncertainty of RSSL was estimated to be plus or minus 5.7 mg/kg at 167 mg/kg for a spiked sausage sample, calculated as 2 x standard deviations. The standard deviation was found to be 1.7 per cent. The results were adjusted for recovery. Test samples with sulphur dioxide contents close to this value would have a similar uncertainty, but this cannot necessarily be applied to samples with an appreciably different sulphur dioxide content. The uncertainty for DSS was estimated, using data from previous analyses of meat products, to be plus or minus 17 mg/kg at the 300 mg/kg concentration level for the volumetric method and expanded uncertainty of 25 mg/kg for the gravimetric method.

The limit of quantification (LOQ) for the RSSL method was 5 mg/kg. The limit of detection (LOD) for the Durham volumetric method was 10 mg/kg and the LOQ was 45 mg/kg. The LOD for the Durham gravimetric method was 15 mg/kg and the LOQ was 50 mg/kg. All reported results were corrected for recovery by both laboratories.

## **Reporting**

Brand names have been reported as this survey was carried out in accordance with the guidelines for reporting survey results published in the *Guidelines for Food Standards Agency Technical Surveys* in October 2003<sup>3</sup>.

## **Results, Interpretation and Action**

The levels of sulphur dioxide in all the tested samples were within the statutory limits. This is 350 mg/kg for lime and lemon juice and concentrates based on fruit juice containing not less than 2.5 per cent barley (barley water) and 250 mg/kg for other concentrates based on fruit juice or comminuted fruit. Results are given in Tables 1 and 2.

One squash sample, 211C003, had levels of sulphur dioxide at 268 mg/kg. As this was above the permitted 250 mg/kg a second bottle of the same sample, was sent to DSS for confirmatory analysis, where it was determined to be 210 mg/kg. To rule out variation between the two bottles and to clarify whether samples met the maximum permitted limits, the laboratories swapped portions of the squash. In addition to analysing the swapped portion of squash, the laboratories also re-analysed their original bottle.

A further sample, 211C015, had a level of 254 mg/kg and was sent for confirmatory analysis at DSS, where it was determined to be 235 mg/kg by the volumetric method and 240 mg/kg by the gravimetric method. Results of all confirmatory analyses are given in Table 4.

Once measurement of uncertainty of the analytical methods used by the two laboratories were taken into account both sample 211C003 and 211C015 were considered to be within the maximum permitted levels. Measurement of uncertainty is used to determine whether a sample is in compliance with the statutory limit. Thus, if the analytical value less the

uncertainty is still greater than the statutory limit, it may be said that the sample is in excess of that statutory limit beyond any reasonable doubt. As such, no action was taken on the concerned samples. The differences between the results obtained by the two laboratories are consistent with an analysis such as this.

Sulphites are chemically unstable in foods. In particular sulphites can convert to sulphur dioxide gas, which can leave the product when the packaging is opened. Previous studies<sup>4,5</sup> have also shown that sulphur dioxide levels reduce during storage and these levels are likely to reduce further due to frequent opening and closing of the bottle. However this is yet to be fully researched and in order to investigate this further the Agency is planning to conduct a research project looking at the fate of sulphites during storage and/or cooking of prepared foods. The findings of this research will be used together with the results from this survey to refine intakes estimates for sulphur dioxide, which will be used to inform future EU discussions on maximum permitted levels of these additives in foodstuffs.

## References

1. The Miscellaneous Food Additives Regulations 1995 (Statutory Instrument No 3187, as amended).
2. Scientific Committee for Food, Opinion on sulphur dioxide and other sulphiting agents used as food preservatives. Reports of the Scientific Committee for Food thirty-fifth series, February 1994.
3. Food Standards Agency Guidelines for Food Standards Agency Technical Surveys, October 2003.
4. Pearson.D, A note on losses of vitamin C and sulphur dioxide in Ribena during storage. *Journal of the Association of Public Analysts*. 1975 **13** (2), 76-7.
5. Vidyasagar.K, and Arya.S.S. Degradation of sorbic acid in fruit squashes and fish paste. *Journal of Food Technology*. 1984 **19**, 447-454

## **Further information**

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This is the final report of this survey. Copies of it have been placed on the Agency's website and in the library in Aviation House, 125 Kingsway, London WC2B 6NH.

**Table 1: Sample details and results. Results given in mg/kg.**

SAMPLE CODE	PRODUCT AS DESCRIBED	BRAND	BEST BEFORE	BATCH CODE	UNIT VOLUME	FRUIT CONTENT	NAME OF RETAIL OUTLET	DATE SAMPLE PURCHASED	SUGGESTED DILUTION FACTOR	SO2 CONTENT IN CONCENTRATE (mg/kg)
211C001	Blackcurrant	Mi Wadi	31 JUL 04	L3297	1 litre	2%	West End Store	16.12.03	1:4	110
211C002	Orange Drink	Super Valu	24OCT04	L32971	1 litre	10%	Super Valu	16.12.03	1:4	127
211C003	Lemon Drink	Super Valu	31OCT04	L33042	1 litre	10%	Super Valu	16.12.03	1:4	268
211C004	Orange (No Added Sugar)	Mi Wadi	30 JUN 04	L3276	1 litre	16%	Super Valu	16.12.03	1:4	25
211C005	Orange & Pineapple	Mi Wadi	31 JUL 04	L3294	1 litre	14%	Super Valu	16.12.03	1:4	91
211C006	Blackcurrant Drink	Super Valu	22OCT04	L32953	1 litre	5%	Super Valu	16.12.03	1:4	189
211C007	Fruit & Barley Peach (No Added Sugar)	Robinsons	OCT04	N14 3274	1 litre	17%	Tesco	16.12.03	1:4 Add extra water for toddlers	74
211C008	Tropical Squash (No Added Sugar)	Tesco	AUG 04	3330 D4	1 litre	13.5%	Tesco	16.12.03	1:4 Add extra water for toddlers	196,197

**Table 1: Sample details and results. Results given in mg/kg (cont'd).**

SAMPLE CODE	PRODUCT AS DESCRIBED	BRAND	BEST BEFORE	BATCH CODE	UNIT VOLUME	FRUIT CONTENT	NAME OF RETAIL OUTLET	DATE SAMPLE PURCHASED	DILUTION SQUASH: WATER	SO2 CONTENT IN CONCENTRATE (mg/kg)
211C010	Lemon & Lime Squash	Tesco	Nov 04	3305 D4	1 litre	10%	Tesco	16.12.03	1:4 Add extra water for toddlers	139
211C011	High Juice White Grape & Peach Squash	Asda	JUL 04	D7 3304	1 litre	50%	Asda	01.12.03	1:4 Add extra water for toddlers	133
211C013	Fruity Apple and Strawberry Hi-Juice (No Added Sugar)	Asda	APR 04	D7 3296	1 litre	50%	Asda	02.12.03	1:4 Add extra water for toddlers	163
211C014	Spiced Apple & Blackcurrant Winter Special Edition	Robinsons	SEP 04	N11 3268	1 litre	50%	Asda	01.12.03	1:4	86
211C015	Special R Orange (No Added Sugar)	Robinsons	NOV 04	N12 3325	1 litre	11%	Iceland	03.12.03	1:4 Add extra water for toddlers	254
211C016	Special R Lemon (No Added Sugar)	Robinsons	OCT 04	N14 3295	1 litre	12%	Iceland	03.12.03	1:4 Add extra water for toddlers	158,157
211C018	Strawberry Drink (No Added Sugar)	Co-op	OCT 04	3293 D4	1 litre	11%	Co-op	03.12.03	1:4 Use half the quantity of concentrate for small children	149
211C019	Pink Grapefruit Fruit & Barley (No Added Sugar)	Co-op	MAR 04	D7 3173	1 litre	12%	Co-op	03.12.03	1:4 Use half the quantity of concentrate for small children	4,8

**Table 1: Sample details and results. Results given in mg/kg (cont'd).**

SAMPLE CODE	PRODUCT AS DESCRIBED	BRAND	BEST BEFORE	BATCH CODE	UNIT VOLUME	FRUIT CONTENT	NAME OF RETAIL OUTLET	DATE SAMPLE PURCHASED	DILUTION SQUASH: WATER	SO2 CONTENT IN CONCENTRATE (mg/kg)
211C020	Blackcurrant Juice Drink Toothkind	Ribena	AUG 2004	483C	600ml	34%	Somerfield	09.01.04	1:5, Not specifically designed for young children (1-3 years), if given max. 1 serving a day. Not suitable for babies under 1 year	171
211C021	Fruit & Barley Summer Fruits (No Added Sugar)	Robinsons	NOV04	N14 3316	1 litre	15%	Asda	12.12.03	1:4 Add extra water for toddlers	114
211C022	Special R Orange & Pineapple (No Added Sugar)	Robinsons	DEC04	N14 3337	1 litre	13%	Asda	12.12.03	1:4 Add extra water for toddlers	232
211C023	High Juice Pink Grapefruit	Robinsons	SEP04	N11 3254	1 litre	51%	Asda	12.12.03	1:4	143
211C024	Original Cranberry Juice Drink	Ocean Spray	JUN 04	D7 3267	1 litre	40%	Asda	12.12.03	1:4 Add extra water for toddlers	99
211C026	Orange, Lemon & Pineapple Drink (No Added Sugar)	Co-op	JUL 04	3301 D4	1 litre	10%	Co-op	13.12.03	1:4 Use half quantity of concentrate for small children	172,177
211C027	Tropical Fruit Drink (No Added Sugar)	Safeway	AUG 04	B 3306 D4	1 litre	15%	Safeway	14.12.03	1:4 Add extra water for toddlers	61

**Table 1: Sample details and results. Results given in mg/kg (cont'd).**

SAMPLE CODE	PRODUCT AS DESCRIBED	BRAND	BEST BEFORE	BATCH CODE	UNIT VOLUME	FRUIT CONTENT	NAME OF RETAIL OUTLET	DATE SAMPLE PURCHASED	DILUTION SQUASH: WATER	SO2 CONTENT IN CONCENTRATE (mg/kg)
211C028	Apple & Blackcurrant Drink	Safeway	OCT 04	B 3274 D4	1 litre	10%	Safeway	14.12.03	1:4 Add extra water for toddlers	173,173
211C029	High Juice Cloudy Apple	Safeway	AUG 04	3311	1 litre	50%	Safeway	14.12.03	1:4 Add extra water for toddlers	83
211C030	Whole Orange Drink (No Added Sugar)	Safeway Savers	AUG 04	B 3310 D4	1 litre	5%	Safeway	14.12.03	1:4 Add extra water for toddlers	200
211C031	Orange Squash	Waitrose	OCT 04	D7 L304	1 litre	20%	Waitrose	11.12.03	1:4 Add extra water for toddlers	73,71
211C033	High Juice Orange & Mango Squash	Waitrose	SEP 04	D7 L257	1 litre	50%	Waitrose	11.12.03	1:4 Add extra water for toddlers	201
211C034	Lime Juice Cordial	Waitrose	OCT 04	D7 L295	1 litre	25%	Waitrose	11.12.03	1:4	180
211C035	Mixed Fruit Squash	Kia Ora	FEB 04	A S 043SC7	1 litre	14%	b4 Convenience Store	11.12.03	1:4 Add extra water for toddlers	6

**Table 1: Sample details and results. Results given in mg/kg (cont'd).**

SAMPLE CODE	PRODUCT AS DESCRIBED	BRAND	BEST BEFORE	BATCH CODE	UNIT VOLUME	FRUIT CONTENT	NAME OF RETAIL OUTLET	DATE SAMPLE PURCHASED	DILUTION SQUASH: WATER	SO2 CONTENT IN CONCENTRATE (mg/kg)
211C036	Whole Orange Squash	Best-in	MAY 04	C 3134	1 litre	10%	Euro Food & Wine	11.12.03	1:4 Add extra water for toddlers	162
211C037	Whole Orange Squash	Londis	AUG 04	3243 D4	1 litre	10%	Pop-In	11.12.03	1:4 Add extra water for toddlers	31
211C038	Orange Lemon Pineapple Squash (No Added Sugar)	Londis	MAY 04	3229 D4	1 litre	13%	Pop-In	11.12.03	1:4 Add extra water for toddlers	161
211C042	Orange Drink (No Added Sugar)	Nisa Today's Value	JUL 04	3279 D4	1 litre	10%	Budgens	11.12.03	1:4 Add extra water for toddlers	206
211C043	Whole Orange Squash	Budgens	OCT 04	3288 D4	1 litre	10%	Budgens	11.12.03	1:4 Add extra water for toddlers	43,45
211C044	Orange Squash	Somerfield	SEP 04	3255 D4	1 litre	10%	Somerfield	09.01.04	1:4 Add extra water for toddlers	60
211C046	Original Apple	Robinsons	NOV 04	N12 3321	1 litre	10%	Tesco	09.01.04	1:4 Add extra water for toddlers	67
211C047	Forest Fruits High Juice	Robinsons	NOV 04	N11 3324	1 litre	50%	Tesco	09.01.04	1:4	94

**Table 1: Sample details and results. Results given in mg/kg (cont'd).**

SAMPLE CODE	PRODUCT AS DESCRIBED	BRAND	BEST BEFORE	BATCH CODE	UNIT VOLUME	FRUIT CONTENT	NAME OF RETAIL OUTLET	DATE SAMPLE PURCHASED	DILUTION SQUASH: WATER	SO2 CONTENT IN CONCENTRATE (mg/kg)
211C049	Elderflower Cordial	Thorncroft	OCT 05	A3294	37.5cl	None declared	Sainsbury's	09.01.04	1:12	48,49
211C050	Blackcurrant Juice Drink	Ribena Original	NOV 2004	483E	600ml	22%	Sainsbury's	15.01.04	1:5 Not designed for young children (1-3 years), if given max. 1 serving a day. Not suitable for babies under 1 year	60
211C051	Tropical Fruit Squash (No Added Sugar)	Sainsbury's	SEP 04	3351 D4	1 litre	10%	Sainsbury's	09.01.04	1:4 Add extra water for toddlers	189
211C052	Orange and Mango Squash (No Added Sugar)	Sainsbury's	AUG 04	3331 D4	1 litre	10%	Sainsbury's	09.01.04	1:4 Add extra water for toddlers	188
211C053	Orange Squash (No Added Sugar)	Sainsbury's	SEP 04	3343 D4	1 litre	10%	Sainsbury's	09.01.04	1:4 Add extra water for toddlers	177
211C054	Orange, Lemon and Pineapple Squash (No Added Sugar)	Sainsbury's	AUG 04	3306 D4	1 litre	10%	Sainsbury's	09.01.04	1:4 Add extra water for toddlers	175
211C055	Orange Squash (No Added Sugar)	Sainsbury's Low Price	AUG 04	3330 D4	1 litre	5%	Sainsbury's	09.01.04	1:4 Add extra water for toddlers	192

**Table 1: Sample details and results. Results given in mg/kg (cont'd).**

SAMPLE CODE	PRODUCT AS DESCRIBED	BRAND	BEST BEFORE	BATCH CODE	UNIT VOLUME	FRUIT CONTENT	NAME OF RETAIL OUTLET	DATE SAMPLE PURCHASED	DILUTION SQUASH: WATER	SO2 CONTENT IN CONCENTRATE (mg/kg)
211C056	Orange Squash (No Added Sugar)	Sunquen	OCT 04	L4 3293	1 litre	10%	Aldi	24.11.03	1:4	36
211C057	High Juice Apple Squash	Hyberry	SEP 04	L4 3266	1 litre	50%	Aldi	24.11.03	1:4	65
211C058	Lemon & Lime Squash (No Added Sugar)	Morrisons	JUL 04	L4 3285	1 litre	10%	Morrisons	24.11.03	1:4 Add extra water for toddlers	226
211C059	Elderflower Cordial	Belvoir	AUG 2005	240	37.5 cl	None Declared	Morrisons	24.11.03	1:10	47
211C060	High Juice Forest Fruits	Morrisons	OCT 04	L4 3279	1 litre	50%	Morrisons	24.11.03	1:4	90,90
211C061	Fruit & Barley Orange (No Added Sugar)	Morrisons	JUN 04	D7 3269	1 litre	11.5%	Morrisons	24.11.03	Dilute to taste Add extra water for toddlers	39
211C062	Blackcurrant Juice Drink (No Added Sugar)	C-Vit	JUN 2004	363B	1 litre	15%	Morrisons	24.11.03	1:4 Toddlers maximum 1 serving a day	130,130
211C063	Lime Juice Cordial	Morrisons	OCT 04	L4 3281	1 litre	25%	Morrisons	24.11.03	1:4 Add extra water for toddlers	195

**Table 1: Sample details and results. Results given in mg/kg (cont'd).**

SAMPLE CODE	PRODUCT AS DESCRIBED	BRAND	BEST BEFORE	BATCH CODE	UNIT VOLUME	FRUIT CONTENT	NAME OF RETAIL OUTLET	DATE SAMPLE PURCHASED	DILUTION SQUASH: WATER	SO2 CONTENT IN CONCENTRATE (mg/kg)
211C064	Spiced Berry Cordial	Bottlegreen	OCT 04	135303115	50 cl	40%	Tesco	25.11.03	1:8 Hot water	10
211C065	Low Calorie Orange Drink	Tesco Value	AUG 04	3307 D4	1 litre	5%	Tesco	25.11.03	1:4 Add extra water for toddlers	191
211C066	Fruit & Barley Orange (No Added Sugar)	Tesco	JUL 04	D7 3289	1 litre	21%	Tesco	25.11.03	1:4 Add extra water for toddlers	98
211C067	High Juice Pink Grapefruit Squash (No Added Sugar)	Tesco	OCT 04	D7 3282	1 litre	50%	Tesco	25.11.03	1:4 Add extra water for toddlers	193
211C068	Honeydew Melon, Grape & Lychee Cordial	Tesco Finest	AUG 04	111203220	500 ml	50%	Tesco	25.11.03	1:4 Add extra water for toddlers	24
211C069	Apple & Blackcurrant Squash (No Added Sugar)	Tesco	AUG 04	3314 D4	1 litre	10%	Tesco	25.11.03	1:4 Add extra water for toddlers	75
211C070	Orange, Lemon & Pineapple Squash	Jucee (Princes)	JUN 04	D6 3272	2 litre	10%	Deans Food & Wine	25.11.03	1:4 Add extra water for toddlers	131
211C071	High Juice Pineapple	Safeway	JUL 04	3280	1 litre	50%	Safeway	25.11.03	1:4 Add extra water for toddlers	74

**Table 1: Sample details and results. Results given in mg/kg (cont'd).**

SAMPLE CODE	PRODUCT AS DESCRIBED	BRAND	BEST BEFORE	BATCH CODE	UNIT VOLUME	FRUIT CONTENT	NAME OF RETAIL OUTLET	DATE SAMPLE PURCHASED	DILUTION SQUASH: WATER	SO2 CONTENT IN CONCENTRATE (mg/kg)
211C072	Apple & Blackcurrant Squash	Costcutter	OCT 04	3278 D4	1 litre	10%	Costcutter	25.11.03	1:4 Add extra water for toddlers	172
211C073	Apple & Blackcurrant Squash (No Added Sugar)	Spar	JUL 04	3277 D4	1 litre	10%	A & S Stores	24.11.03	1:4 Add extra water for toddlers	64
211C074	Blackcurrant High Juice	Marks & Spencer	04/03/04	DU 05/02/04 j	1 litre	50%	Marks & Spencer	25.11.03	1:4 Add extra water for toddlers	123
211C075	Florida Pink Grapefruit High Juice	Marks & Spencer	04/03/04	DU 05/02/04 h D7	1 litre	50%	Marks & Spencer	25.11.03	1:4 Add extra water for toddlers	104

**Table 2: Sample details and results of lemon, lime and barley water samples with more than 2.5% barley.  
Results given in mg/kg.**

SAMPLE CODE	PRODUCT AS DESCRIBED	BRAND	BEST BEFORE	BATCH CODE	UNIT VOLUME	FRUIT CONTENT	NAME OF RETAIL OUTLET	DATE SAMPLE PURCHASED	SUGGESTED DILUTION FACTOR	SO2 CONTENT IN CONCENTRATE (mg/kg)
211C009	Lemon	Jif	SEP 04	3251	250ml	None declared	Super Valu	16.12.03	None declared	240,241
211C012	Lemon Barley Water	Robinsons	NOV 04	N14 3311	1 litre	17%	Asda	01.12.03	1:4 Add extra water for toddlers	256
211C017	Orange Barley Water	Robinsons	SEP 04	N14 3268	1 litre	17%	S R Bailey	03.12.03	1:4 Add extra water for toddlers	251
211C025	Lemon Juice	Asda Smartprice	06.10.04	L3279	500ml	None declared	Asda	12.12.03	None declared	327,348
211C032	Lemon Juice	PLJ	MAY 2004	L3262	500ml	None declared	Waitrose	11.12.03	At least 1:4	195
211C039	Lemon Juice	White Pearl	05.06.2004	None declared	250ml	None declared	Rina News	11.12.03	None declared	236
211C040	Lemon Juice	Waitrose	JUL 2004	L3303	125ml	25%	Waitrose	11.12.03	None declared	267
211C041	Lime Juice	Waitrose	APR 2004	L3192	125ml	25%	Waitrose	11.12.03	None declared	20
211C045	Lemon Juice	Tesco	06 11 04	L3310 A	250ml	None declared	Tesco	09.01.04	None declared	208,209
211C048	Lemon Juice	Sainsbury's	11 12 04	L3345 C	250ml	None declared	Sainsbury's	09.01.04	None declared	191

### Table 3: Supplementary analysis by an independent laboratory

Notes: Analytical data (mg/kg) are for aliquots of the same samples analysed independently by Durham Scientific Services & RSSL.

		READING SCIENTIFIC SERVICES LTD	DURHAM SCIENTIFIC SERVICES	
SAMPLE CODE	PRODUCT AS DESCRIBED	VOLUMETRIC (mg/kg)	VOLUMETRIC (mg/kg)	GRAVIMETRIC (mg/kg)
211C004	Orange (No Added Sugar)	25	12,18	12,18
211C007	Fruit & Barley Peach (No Added Sugar)	74	47	58
211C010	Lemon & Lime Squash	139	111	134
211C023	High Juice Pink Grapefruit	143	123	146
211C024	Original Cranberry Juice Drink	99	99	128
211C025	Lemon Juice	327,348	327,350	356,368
211C026	Orange, Lemon & Pineapple Drink (No Added Sugar)	172,177	128	158
211C028	Apple & Blackcurrant Drink	173,173	128	128

**Table 4: Confirmatory data for samples with sulphur dioxide near the permitted limit of 250 mg/kg**

Samples were those that contained sulphur dioxide close to the legal limit for sulphur dioxide. The results of confirmatory analysis are shown, together with results of the original assays. Results are in mg/kg.

Sample code	Analysis	Reading Scientific Services Ltd (RSSL)	Durham Scientific Services (DSS)	
		Volumetric (mg/kg)	Volumetric (mg/kg)	Gravimetric (mg/kg)
211C003	Original	268	x	x
	Confirmatory - bottle sent to DSS	x	145	210
Laboratories swapped portions of squash from their original bottles of sample 211C003	RSSL Bottle	243,244 <sup>a</sup>	210,205 <sup>b</sup>	235,245 <sup>c</sup>
	DSS Bottle	242,249 <sup>a</sup>	210 <sup>b</sup>	230 <sup>c</sup>
211C015	Original	254	x	x
	Confirmatory - bottle sent to DSS	241,240	235	240

<sup>a</sup> Analysed in same analytical run at RSSL

<sup>b</sup> Analysed in same analytical run at DSS using volumetric determination

<sup>c</sup> Analysed in same analytical run at DSS using gravimetric determination