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ANTIMONY, ARSENIC, BROMATE AND NICKEL CONTENTS OF BOTTLED WATER

1. Key Facts

- Bottled water legislation is changing. In December 2003, a reduction in the permitted levels of antimony, arsenic, bromate and nickel will come into force to better protect consumers.
- The objective of this survey was to determine levels of these four substances in all types of bottled waters currently sold in the UK (natural mineral water, spring water and bottled drinking water).
- Of the 161 samples that were collected, none contained antimony or nickel at a concentration above either the current or future limits.
- Allowing for measurement uncertainty, no sample contained arsenic above the current limit, but seven samples exceeded the future limit.
- A single sample exceeded the future limit for bromate.
- Where samples exceeded the future limit, manufacturers have been informed to enable them, where appropriate, to take remedial action.
- Although no waters in the survey contained levels of antimony, arsenic or nickel that exceed current statutory limits, the limits are being reduced in 2003 in line with the latest scientific advice. In the meantime, consumers may wish to use the survey results as a guide when planning purchases.

Summary

2. Legislation, currently in draft, which sets new limits for antimony, arsenic, bromate and nickel in bottled waters will come into force on 25th December 2003.^{1,2} This report describes the findings of a survey carried out in April, looking at bottled waters collected

between October 2001 and February 2002, to investigate the levels of antimony, arsenic, bromate and nickel in the water and to determine compliance with current and proposed limits.

3. The maximum concentrations of antimony and nickel detected were 4.2 and 9.6 micrograms per litre, respectively. Both of these values are below existing and future limits.
4. The concentrations of arsenic ranged from below the limit of detection up to 59.7 micrograms per litre. This highest value was found in a single sample. All remaining samples contained less than 25 micrograms per litre, well below the current limit (50 micrograms per litre). Seven samples, when taking measurement uncertainty into account, contained arsenic at levels greater than 10 micrograms per litre, the limit that will apply from 25th December 2003.
5. As with the other parameters, bromate analysis was carried out at two laboratories. Although two samples contained measurable levels of bromate at both laboratories, a further 8 samples contained bromate at above the limit of detection in measurements from one laboratory. A single sample was identified as being above the future limit of 10 micrograms per litre.
6. No current levels were exceeded. Where a future limit was exceeded, the manufacturer was informed. The manufacturers concerned did not want to comment on the data.

Background

7. The Natural Mineral Water, Spring Water and Bottled Drinking Water Regulations 1999 set maximum limits for antimony (10 micrograms per litre), arsenic (50 micrograms per litre) and nickel (50 micrograms per litre).¹ These limits apply to all three categories of bottled waters. Currently, bottled waters are not subject to a limit for bromate.
8. In 1998, Council Directive 98/83 on water for human consumption was published and set new maximum limits for antimony (5.0 micrograms per litre), arsenic (10 micrograms per litre), bromate (10 micrograms per litre) and nickel (20 micrograms per

litre).² The Water Supply (Water Quality) Regulations 2000 transpose this Directive as it applies to tap water in the UK³, whilst new draft legislation⁴ amending The Natural Mineral Water, Spring Water and Bottled Drinking Water Regulations 1999 will transpose the Directive as it applies to bottled waters. Both the public water supply legislation and the new bottled water amending legislation will require water to meet these new limits from 25th December 2003.

9. To remain in step with the introduction of the future limits for tap water, the new limits for the parameters in bottled water will apply from the 25th December 2003. Given this timing, the Food Standards Agency decided to check the levels of these four substances in bottled waters currently on the UK market.

Methodology

Samples

10. 161 samples (104 natural mineral water, 44 spring water and 13 bottled drinking water) were collected between 10th October 2001 and 15th February 2002, and represented 101 brands. For some brands, more than one sample was collected to ensure that sampling took account of the market share for the main brands of bottled water sold in the UK. Of the samples produced in the UK, sampling matched the geographical contribution to UK production. 40% of the samples were supermarket own brands. Where possible, each sample comprised two bottles (each at least 330 ml) of water with the same batch code.

Methods

11. The levels of antimony, arsenic, bromate and nickel measured in this survey were extremely low and required very sensitive analytical methods. Additional confidence in the results was gained by employing two laboratories to analyse the same samples. The laboratories, Compagnie Intercommunale Bruxelloise des Eaux (CIBE) in Brussels and Harwell Scientifics in Oxfordshire, were able to meet the required analytical performance, in some cases using different methods. For the majority of samples, one bottle was analysed by each laboratory. CIBE analysed each sample in duplicate whilst Harwell analysed one in every twelve samples in duplicate. In addition, each laboratory ran blank samples, a calibration curve and internal reference materials for each analyte. These were all used in the statistical analysis of the data.

1. Samples analysed by CIBE, used hydride formation, atom generation and fluorescence detection to quantify levels of arsenic and antimony. Nickel analysis was carried out by atomisation to inductively coupled plasma, and fluorescence detection. Bromate analysis was carried out using ion chromatography followed by post-column transformation to bromine and UV absorption at 267nm. In contrast, samples were analysed at Harwell by inductively coupled plasma-mass spectrometry to quantify levels of arsenic, antimony and nickel. A similar method to that used by the Brussels laboratory was used by Harwell to quantify bromate. The existing and new limits for the four parameters are presented in Table 1 of Annex 1, together with the limits of detection for each method. These limits of detection met the requirements of Directive 98/83.²

Statistical analysis of results

13. Linear regression was used to take account of differences between measured values and actual values obtained for control samples in each laboratory. The coefficient of variation was used to estimate the typical error associated with each sample. Reference to an appropriate T-distribution was made to determine measurement uncertainty values. The reported mean (Table 3) value differs from the actual mean because it takes into account between-laboratory and within-laboratory variation. The measurement uncertainty (U) associated with the reported mean is the 95% confidence limit.

Results

14. The results (reported means and measurement uncertainty) of the analyses are presented in Tables 2 and 3. The sample code, which was randomly assigned to the sample by the collection company, relates to the full sample description located on the [Food Standards Agency website \(www.food.gov.uk/science/surveillance/foodsurvprog#h_6\)](http://www.food.gov.uk/science/surveillance/foodsurvprog#h_6). The absence of a particular brand from the Table indicates only that the brand was not included in the samples collected. For some brands, more than one sample was collected. The mean value for the brand is also given, immediately after the values for the individual samples. Where one or more of the recorded values for a sample were less than the

limit of detection, the reported mean value for that sample is less than the limit of detection.

15. Generally, the values obtained by one laboratory matched those obtained by the other, despite, in some instances, different methods being employed. This is illustrated in Table 4 which gives the value for each replicate measured at each laboratory for those samples where antimony, arsenic and nickel were measured in duplicate at both laboratories. The values for antimony, arsenic and nickel measured by each laboratory for each sample are very similar, but there is more between-laboratory variability with bromate (Table 3). With antimony, arsenic and nickel, the reported mean is the same as the actual mean (mean of replicates) for most of the samples analysed (see statistical analysis in methodology).

Significance of results

16. All results below refer to reported means in Table 2 (or Table 3 for bromate). Measurement uncertainty (U) is reported as 95% confidence limits.

Antimony

17. Antimony was detected in 87 samples, but always at levels below both the existing and future limits (10 and 5 µg/l, respectively).

Arsenic

18. Arsenic was found in 78 samples, the highest concentration being 59.7 micrograms per litre. However, taking into account the measurement uncertainty, the reported mean was not above the existing limit (50 micrograms per litre), but would be above the future limit (10 micrograms per litre). The reported means for all other samples were below the existing limit, but a further six samples would exceed the future limit (taking into account measurement uncertainty).

Bromate

19. There is no current limit for bromate in bottled waters. However, bromate was detected in one sample at a level that would exceed the future limit (10 micrograms per litre), taking into account measurement uncertainty. Bromate analysis gave the least consistent inter-laboratory data (Table 3), however, this may be because of the proximity of the measured values and the limit of detection (see Table 1).

Nickel

20. Nickel was detected in 6 samples, but always at levels below both the existing and future limits (50 and 20 µg/l, respectively).

Interpretation

21. The results of this survey indicate that none of the brands sampled exceeded current limits for antimony, arsenic and nickel (taking into account measurement uncertainty). However, eight samples exceeded the future limit for either arsenic or bromate.
22. It should be emphasised that the samples were collected for this survey during a five month period in 2001-2002 and may not represent products on the market after 25th December 2003 (when the future limits come into force).
23. Arsenic occurs naturally in some underground sources of water that percolate through rock strata containing arsenic ores. Directive 80/777 (as amended by Directive 96/70) permits the removal of arsenic from natural mineral and spring waters using ozone treatment, but the conditions of ozone use have not yet been agreed. In some cases, the use of ozone to ensure compliance with the future arsenic limit could create difficulties in complying with the new bromate limit. This is because bromate may be present as a result of ozone treatment of bromide-containing waters. Bromate formation can also arise when bottled drinking waters are disinfected with ozone.
24. Although no current limits were exceeded, manufacturers' of products that exceeded future limits were invited to comment. No comments have been received.

Conclusions

25. None of the samples contained levels of antimony, arsenic, bromate or nickel which exceeded the current limits when measurement uncertainty was accounted for. All waters currently on the UK market would meet the future limits for antimony and nickel, and the majority would meet the future limits for arsenic and bromate. Companies producing bottled waters that contain levels of arsenic or bromate that

would exceed future limits have been informed of the results obtained for their samples.

References

- 1 *The Natural Mineral Water, Spring Water and Bottled Drinking Water Regulations 1999* (S.I. No. 1999/1540).
- 2 Council Directive 98/93/EC of 3 November 1998 on the quality of water intended for human consumption.
- 3 *The Water Supply (Water Quality) Regulations 2000* (S.I. No. 2000/3184).
- 4 *The Natural Mineral Water, Spring Water and Bottled Drinking Water (Amendment) (England) Regulations 2002*.

Further Information

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Annex 1

Table 1 gives the existing and new limits for the parameters being measured, together with the limit of detection (LOD) for each parameter in each laboratory

	Antimony	Arsenic	Bromate	Nickel
Existing Limit (µg/l)	10	50	No limit	50
New Limit (µg/l)	5	10	10	20
CIBE LOD (µg/l)	0.1	0.05	0.2	1
Harwell LOD (µg/l)	0.03	0.4	2	0.3

The Natural Mineral Water, Spring Water and Bottled Drinking Water Regulations 1999 lay down no limit for bromate.

CIBE: Compagnie Intercommunale Bruxelloise des Eaux

Harwell: Harwell Scientifics Ltd