

Monitoring trace anion contamination in disk drive components

Edward Kaiser^{a,*}, Jeff Rohrer^a, Faye Campbell^b

^aDionex Corporation, P.O. Box 3603, 1228 Titan Way, Sunnyvale, CA 94088-3603, USA

^bMaxtor Corporation, 500 McCarthy Boulevard, Milpitas, CA 95035, USA

Abstract

Ion chromatography was used to determine trace anionic contamination on the surface of hard disk drive components. These contaminants can have a detrimental effect on device reliability and yield. Disk drive components were soaked in deionized water and these extracts were analyzed for anions. The anions fluoride, acetate, formate, acrylate, methacrylate, chloride, nitrite, bromide, nitrate, benzoate, sulfate, oxalate, phthalate and phosphate were separated on a high-performance anion-exchange column and determined at concentrations less than 1 µg/l with suppressed conductivity detection. The extract solutions were analyzed either by injecting 1 ml or by preconcentrating 5 ml. We evaluated the performance of both methods.

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1. Introduction

Modern disk drives have very close head to disk interfaces, making the cleanliness of the components critical to drive performance and reliability. Anions are one of the possible contaminants of drive components. Ionic contamination can come from several sources: packaging materials, human contact, assembly environment, aqueous rinse solutions, solvents, adhesives and lubricants. For example, the presence of elevated levels of sulfate can indicate the presence of residues from machining oils or mold release agents [1]. Organic anions such as formate, oxalate, acrylate and benzoate come from cleaning agents, adhesives or oils [2].

A comprehensive anion analysis of drive components prior to manufacturing has significantly

reduced the incidence of corrosion and head/disk interface failures. The following anions are routinely monitored: fluoride, chloride, bromide, nitrate, sulfate, and phosphate [3]. Acetate, formate, acrylate, methacrylate, benzoate, and oxalate are also sometimes monitored. When present with moisture, some anions, particularly chloride and sulfate, form dilute concentrations of mineral acids and cause corrosion [4].

Ion chromatography (IC) has been used to determine anions on disk drive components [5,6]. For this study, we used the EG40 potassium hydroxide eluent generator and the continuously regenerated anion trap column (CR-ATC) with the IonPac AS17 anion-exchange column. The EG40 generates high-purity and carbonate-free hydroxide eluents on-line to improve performance for anion determinations at trace levels [7]. The CR-ATC continuously removes anionic contaminants from the eluent without the need for regeneration [8]. The AS17 was used as the anion-exchange column because of its unique ability

*Corresponding author. Tel.: +1-408-481-4217; fax: +1-408-737-2470.

E-mail address: edward.kaiser@dionex.com (E. Kaiser).

to separate all of the anions of interest [9]. Two methods have been used to increase IC sensitivity to below 1 $\mu\text{g/l}$: high volume/direct injection [10] and preconcentration [11]. This article describes the use of these approaches to determine trace anion contaminants in the extracts of disk drive components.

2. Experimental

2.1. Chromatographic system

All analysis was carried out on a Dionex (Sunnyvale, CA, USA) DX-600 ion chromatograph. The system consists of a gradient pump (GP50), a conductivity detector (CD25A), conductivity cell with temperature control (DS-3), a liquid chromatography oven (LC30), EG40 eluent generator, and AS40 autosampler. A personal computer equipped with Dionex PeakNet 6 chromatography software was used for data acquisition and instrument control.

All columns used in this study were manufactured by Dionex Corporation. For the analytical separation, an IonPac AG17 guard column (50 \times 2 mm) and IonPac AS17 (250 \times 2 mm) analytical column were used. An IonPac trace anion concentrator TAC-LP1 (35 \times 4 mm) was used for preconcentration and configured as shown in Fig. 1. A 2-mm anion self-regenerating ultra suppressor (ASRS) from Dionex operated in the recycle mode was used to reduce the

conductivity of the eluent¹. The tubing used to connect the chromatographic components was 0.005-in. I.D. (0.125-mm) PEEK (polyether ether ketone). The Dionex CR-ATC continuously regenerated anion trap column was used to remove anionic contaminants in the eluent. Tables 1 and 2 list the chromatographic conditions for the 1-ml direct injection method and the 5-ml preconcentration method, respectively.

The packing material for the IonPac AG17 and AS17 is composed of a highly crosslinked microporous core with an anion-exchange latex attached to the surface. The substrate is a 10.5- μm diameter bead and consists of ethylvinylbenzene crosslinked with 55% divinylbenzene. The surface anion-exchange layer of the IonPac AS17 consists of 75-nm diameter particles bonded to the substrate. The packing material for the IonPac TAC-LP1 low-pressure concentrator column consists of 18- μm diameter particles of ethylvinylbenzene crosslinked with 55% divinylbenzene. The surface anion-exchange layer of the TAC-LP1 consists of 85-nm diameter particles bonded to the substrate.

2.2. Chemicals, solutions and samples

Reagent grade chemicals were used for standard preparation. Deionized water with a specific resistance of 18 $\text{M}\Omega\text{-cm}$ or greater from a deionized water purification system was used to prepare all eluents, reagents, and standards. Anion standards (1000 mg/l) for the analytes of interest were from Dionex or prepared with reagent-grade chemicals from Fisher (Pittsburgh, PA, USA).

Extractions were carried out in 60-ml polymethylpentene (PMP) containers (Nalgene, Rochester, NY, USA) that were presoaked with high purity deionized water. A disk clamp and a disk spacer from a 3.5-in. format desktop disk drive were analyzed by the extraction procedure.

2.3. Extraction procedure

The following procedure was used for the collec-

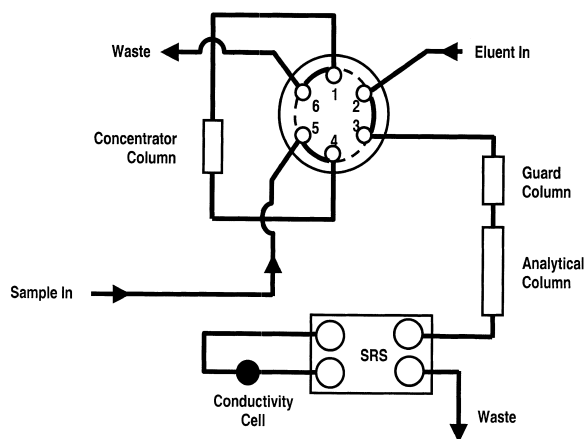


Fig. 1. Instrumentation configuration for preconcentration.

¹PeakNet, IonPac, and ASRS are registered trademarks of Dionex Corporation.

Table 1
Chromatographic conditions for 1-ml direct injection method

Guard column	IonPac AG17 (50×2 mm)				
Analytical column	IonPac AS17 (250×2 mm)				
Eluent	Potassium hydroxide (EG40 as the source)				
Temperature	30 °C				
EG40 offset volume	0 µl				
Eluent flow rate	0.5 ml/min				
Detection	Suppressed conductivity, ASRS-ULTRA recycle mode				
ASRS current setting	100 mA				
Sample volume	1 ml				
Gradient program					
Time (min)	Flow (ml/min)	A (%)	Valve	EG40 conc. (mM)	Comments
–5.00	0.50	100	Load	0.3	0.3 mM KOH
–2.4	0.50	100	Load	0.3	Load loop, AS40 on
–0.1	0.50	100	Load	0.3	AS40 off
0.00	0.50	100	Inject	0.3	Inject 0.3 mM KOH
6.00	0.50	100	Inject	0.3	0.3 mM KOH
8.00	0.50	100	Inject	1.0	1.0 mM KOH
19.00	0.50	100	Inject	10.0	10 mM KOH
19.20	0.50	100	Inject	10.0	10 mM KOH
35.80	0.50	100	Load	40.0	40 mM KOH

Table 2
Chromatographic conditions for 5-ml preconcentration method

Guard column	IonPac AG17 (50×2 mm)				
Analytical column	IonPac AS17 (250×2 mm)				
Concentrator column	IonPac TAC-LP1 (35×4 mm)				
Eluent	Potassium hydroxide (EG40 as the source)				
Temperature	30 °C				
EG40 offset volume	0 µl				
Eluent flow rate	0.5 ml/min				
Detection	Suppressed conductivity, ASRS-ULTRA recycle mode				
ASRS current setting	100 mA				
Sample volume	5 ml				
Pump program					
Time (min)	Flow (ml/min)	A (%)	Valve	EG40 conc. (mM)	Comments
–9.00	0.50	100	Inject	0.3	0.3 mM KOH
–6.5	0.50	100	Load	0.3	Load TAC-LP1, AS40 on
–0.1	0.50	100	Load	0.3	AS40 off
0.00	0.50	100	Inject	0.3	Inject, 0.3 mM KOH
6.00	0.50	100	Inject	0.3	0.3 mM KOH
8.00	0.50	100	Inject	1.0	1.0 mM KOH
19.00	0.50	100	Inject	10.0	10 mM KOH
19.20	0.50	100	Inject	10.0	10 mM KOH
35.80	0.50	100	Inject	40.0	40 mM KOH

tion, storage and analysis of samples and standards with polymethylpentene (PMP) containers. The sample container was rinsed and capped three to five times with deionized water. The container was filled to overflowing and capped securely and then soaked for at least 4 h. The container was emptied and refilled with deionized water and capped securely. The container was soaked for an additional 24 h before sample collection, emptied, and rinsed twice with deionized water.

The disk drive part was added into the extraction vessel and filled with 20 ml of deionized water. The sealed container containing the part and deionized water was heated at 85 °C in an oven for 1 h, removed from the oven, and allowed to come to ambient temperature (approximately 1 h). The soak at elevated temperature was intended to be a more thorough extraction of ionic species than a soak at ambient temperature [12]. By extracting for 1 h, it was possible to cool the sample to ambient temperature and analyze in 1 day. This type of test was intended as a worst case scenario given that hard drives are not submerged in hot water. They can be subjected to higher than ambient temperature conditions and may be in high humidity areas.

The following procedure was used for loading samples into autosampler vials. The vials and caps were rinsed with deionized water three to five times by placing them in a large precleaned container and allowing them to soak in deionized water for 4 h at a time. The 5-ml vial was filled with the standard, sample or blank. The autosampler cap (without filter) was inserted into the vial.

The following calculations were used to determine the weight of the anionic contaminants per unit area [13]. The level in the blank is subtracted from that found in the sample:

$$C_s - C_b = C_{s-b} \quad (1)$$

where C_s is the concentration in the sample, C_b is the concentration in the blank and C_{s-b} is the blank-corrected concentration for the sample in ng/ml. To calculate the total weight in nanograms of the ionic species extracted, the extract volume is multiplied by the volume injected:

$$(C_{s-b} \text{ ng/ml}) \times (20 \text{ ml extracted}) = W \quad (2)$$

where W is the weight of the extracted ion in

nanograms. The weight is referenced to the area of the part with the following equation:

$$W/A = X \quad (3)$$

where A is the area of the part in cm^2 and X is the mass per unit area in ng/cm^2 .

3. Results and discussion

3.1. Choice of system components

The microbore format was chosen for the analytical columns and suppressor because it has several advantages. There is a fourfold increase in mass sensitivity for the microbore (2 mm) over the standard bore (4 mm) format with no change in concentration sensitivity. The increased mass sensitivity allows smaller sample volumes to be concentrated and therefore reduces the time per analysis. The microbore format also uses less eluent and produces eluent waste.

The EG40 Eluent Generator enhances ion chromatographic performance for the determination of anions at trace levels [14]. This device electrolytically produces high-purity, carbonate-free, KOH eluents using deionized water as the carrier stream. Gradient separations with carbonate-free hydroxide eluents have negligible baseline shifts, lower background conductivity and are highly precise. This results in better retention time reproducibility and improved signal-to-noise ratios.

The CR-ATC Continuously Regenerated Anion Trap Column was used to remove trace anionic contaminants in the eluent that arise from the source deionized water. This is a high-pressure electrolytically regenerated trap column device that operates continuously without the need for off-line regeneration, thus reducing instrument downtime [8]. This device further minimizes baseline drift during gradient operation.

The IonPac AS17 column was chosen for this analysis because it provides the best selectivity for the analytes of interest to the electronics industry: common inorganic anions, low molecular mass organic acids, acrylate, methacrylate, benzoate, and phthalate. Other anion-exchange columns that were

evaluated did not adequately resolve the target analytes. For instance, the IonPac AS15-5 μm column has very good resolution for the weakly retained organic acids such as acetate, glycolate, and formate from fluoride [15]. However, several of the target analytes are not well resolved using the IonPac AS15-5 μm : acrylate and chloride as well as carbonate, benzoate and methacrylate [9]. The ASRS-ULTRA delivers low background and noise for good sensitivity at trace levels and the DS-3 conductivity cell minimizes the effects of cell drift and temperature fluctuations.

3.2. Method performance

To achieve sensitivity at trace levels, we designed two methods: direct injection of 1 ml of sample and

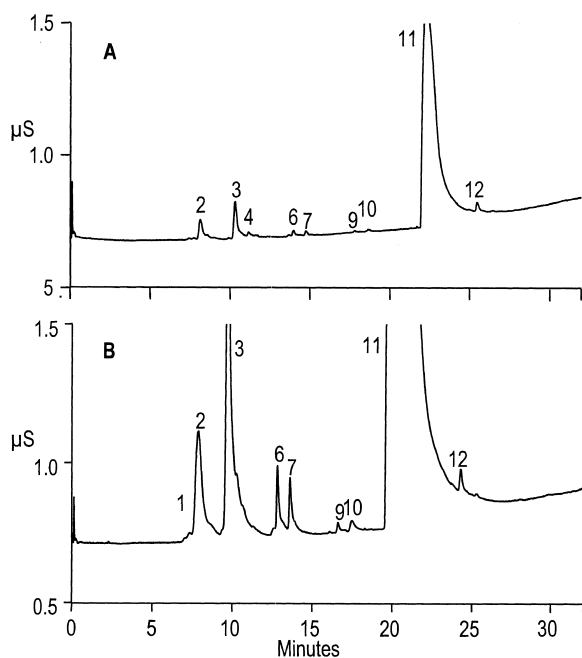


Fig. 2. Deionized water extract system blank. (a) 1-ml Direct injection. Peaks: 2=acetate (1.7 $\mu\text{g}/\text{l}$); 3=formate (3.0 $\mu\text{g}/\text{l}$); 4=acrylate (0.68 $\mu\text{g}/\text{l}$); 6=chloride (0.28 $\mu\text{g}/\text{l}$); 7=nitrite (0.16 $\mu\text{g}/\text{l}$); 9=nitrate (0.10 $\mu\text{g}/\text{l}$); 10=benzoate (0.42 $\mu\text{g}/\text{l}$); 11=carbonate; 12=sulfate (0.64 $\mu\text{g}/\text{l}$); for chromatographic conditions, see Table 1. (b) 5-ml Preconcentration. Peaks: 1=fluoride (0.10 $\mu\text{g}/\text{l}$); 2=acetate (4.6 $\mu\text{g}/\text{l}$); 3=formate (9.5 $\mu\text{g}/\text{l}$); 6=chloride (0.75 $\mu\text{g}/\text{l}$); 7=nitrite (1.2 $\mu\text{g}/\text{l}$); 9=nitrate (0.10 $\mu\text{g}/\text{l}$); 10=benzoate (0.85 $\mu\text{g}/\text{l}$); 11=carbonate; 12=sulfate (0.30 $\mu\text{g}/\text{l}$); for chromatographic conditions, see Table 2.

preconcentration of 5 ml of sample on a TAC-LP1. A method blank was established by subjecting 20 ml of deionized water to all the steps of the extraction procedure. The average concentration for each of the analytes of interest was calculated from seven replicate method blanks. Most analytes were detected below 1 $\mu\text{g}/\text{l}$, except acetate and formate. These average anion concentrations in the method blank were subtracted from the values measured in the extracts of the parts. Representative method deionized water blanks for both approaches are shown in Fig. 2. Determining a blank establishes a starting

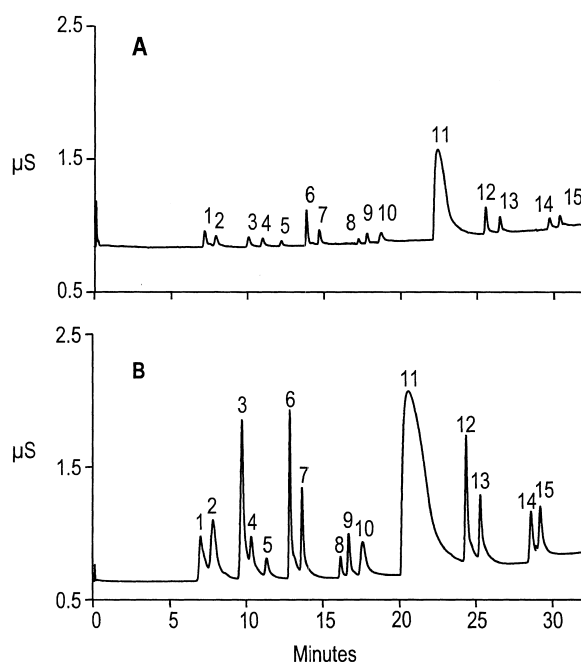


Fig. 3. Trace anion standards. (a) 1-ml Direct injection. Peaks: 1=fluoride (1.1 $\mu\text{g}/\text{l}$); 2=acetate (1.4 $\mu\text{g}/\text{l}$); 3=formate (1.5 $\mu\text{g}/\text{l}$); 4=acrylate (3.1 $\mu\text{g}/\text{l}$); 5=methacrylate (1.5 $\mu\text{g}/\text{l}$); 6=chloride (1.6 $\mu\text{g}/\text{l}$); 7=nitrite (1.3 $\mu\text{g}/\text{l}$); 8=bromide (0.7 $\mu\text{g}/\text{l}$); 9=nitrate (0.9 $\mu\text{g}/\text{l}$); 10=benzoate (5.6 $\mu\text{g}/\text{l}$); 11=carbonate; 12=sulfate (3.1 $\mu\text{g}/\text{l}$); 13=oxalate (2.3 $\mu\text{g}/\text{l}$); 14=phthalate (3.6 $\mu\text{g}/\text{l}$); 15=phosphate (2.9 $\mu\text{g}/\text{l}$); for chromatographic conditions, see Table 1. (b) 5-ml Preconcentration. Peaks: 1=fluoride (1.2 $\mu\text{g}/\text{l}$); 2=acetate (4.8 $\mu\text{g}/\text{l}$); 3=formate (6.6 $\mu\text{g}/\text{l}$); 4=acrylate (1.9 $\mu\text{g}/\text{l}$); 5=methacrylate (2.1 $\mu\text{g}/\text{l}$); 6=chloride (2.6 $\mu\text{g}/\text{l}$); 7=nitrite (3.1 $\mu\text{g}/\text{l}$); 8=bromide (1.0 $\mu\text{g}/\text{l}$); 9=nitrate (1.3 $\mu\text{g}/\text{l}$); 10=benzoate (10 $\mu\text{g}/\text{l}$); 11=carbonate; 12=sulfate (3.8 $\mu\text{g}/\text{l}$); 13=oxalate (2.4 $\mu\text{g}/\text{l}$); 14=phthalate (5.6 $\mu\text{g}/\text{l}$); 15=phosphate (4.1 $\mu\text{g}/\text{l}$); for chromatographic conditions, see Table 2.

point above which trace-level anion determinations can be made.

Both methods begin with a 5-min equilibration at an eluent concentration of 0.3 mM potassium hydroxide. This dilute eluent is used to elute weakly retained ions such as fluoride, acetate, and formate. A linear gradient to a higher KOH concentration is used to separate more strongly retained anions such as sulfate and phosphate. The chromatographic baseline shift during the gradient is typically less than 200 nS when using the EG40. A much larger shift in background conductivity would have been observed with manually prepared eluents [14] and it would have been difficult to reproducibly deliver 0.3 mM KOH. The separations are carried out at 30 °C to provide the best retention time reproducibility during trace analysis. A trace anion standard analyzed by each method is shown in Fig. 3.

There are differences in the chromatographic

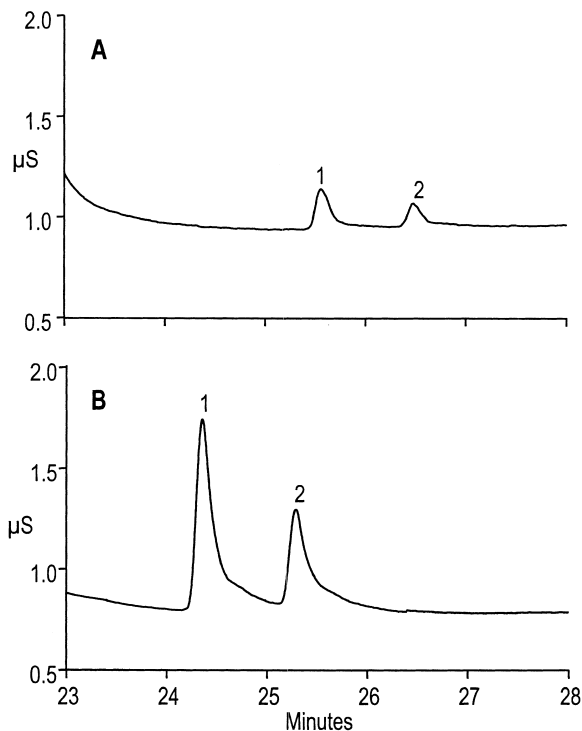


Fig. 4. Trace anion standards. Detail of Fig. 3. (a) 1-ml Direct injection. Peaks: 1 = sulfate ($3.1 \mu\text{g}/\text{l}$); 2 = oxalate ($2.3 \mu\text{g}/\text{l}$); for chromatographic conditions, see Table 1. (b) 5-ml Preconcentration. Peaks: 1 = sulfate ($3.8 \mu\text{g}/\text{l}$); 2 = oxalate ($2.4 \mu\text{g}/\text{l}$); for chromatographic conditions, see Table 2.

performance between the direct injection and the preconcentration methods. To illustrate this, a separation of a sulfate and oxalate standard is shown in Fig. 4. A greater response is observed with the preconcentration technique than with direct injection. This is expected because the 5-ml preconcentration technique loads five times more sample than the 1-ml direct injection technique. Also, observe that the two analytes elute ~ 1.2 min later for the direct injection method compared to the preconcentration method. This is because of the additional time required for the 1-ml sample volume to pass through the void volume of the analytical column set. In the preconcentration method, the sample is loaded off-line

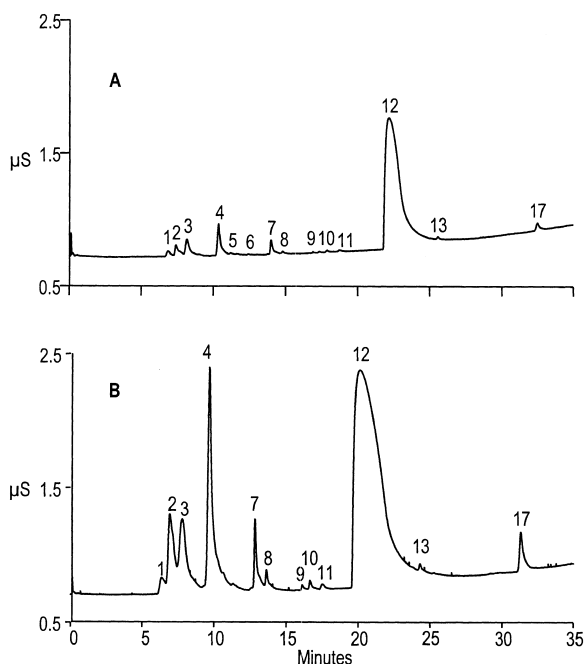


Fig. 5. Analysis of deionized water extract from disk drive spacer. (a) 1-ml Direct injection. Peaks: 1 = unidentified; 2 = fluoride ($1.2 \mu\text{g}/\text{l}$); 3 = acetate ($4.7 \mu\text{g}/\text{l}$); 4 = formate ($4.6 \mu\text{g}/\text{l}$); 5 = acrylate ($0.31 \mu\text{g}/\text{l}$); 6 = methacrylate ($0.15 \mu\text{g}/\text{l}$); 7 = chloride ($1.1 \mu\text{g}/\text{l}$); 8 = nitrite ($0.10 \mu\text{g}/\text{l}$); 9 = bromide ($0.14 \mu\text{g}/\text{l}$); 10 = nitrate ($0.21 \mu\text{g}/\text{l}$); 11 = benzoate ($0.56 \mu\text{g}/\text{l}$); 12 = carbonate; 13 = sulfate ($0.27 \mu\text{g}/\text{l}$); for chromatographic conditions, see Table 1. (b) 5-ml Preconcentration. Peaks: 1 = unidentified; 2 = fluoride ($2.2 \mu\text{g}/\text{l}$); 3 = acetate ($6.7 \mu\text{g}/\text{l}$); 4 = formate ($8.8 \mu\text{g}/\text{l}$); 7 = chloride ($1.4 \mu\text{g}/\text{l}$); 8 = nitrite ($0.85 \mu\text{g}/\text{l}$); 9 = bromide ($0.24 \mu\text{g}/\text{l}$); 10 = nitrate ($0.29 \mu\text{g}/\text{l}$); 11 = benzoate ($0.83 \mu\text{g}/\text{l}$); 12 = carbonate; 13 = sulfate ($0.22 \mu\text{g}/\text{l}$); for chromatographic conditions, see Table 2.

onto the IonPac TAC-LP1 (requires 6 min) and the accumulated anions are rinsed onto the AS17 analytical column set. The peaks are more efficient for the direct injection method because less band broadening occurs when loading sample directly on the analytical column compared to loading onto a concentrator column.

An extract solution of a disk drive spacer was evaluated with both methods. The spacer is fabricated from an aluminum alloy and is used as a spacer between disks. The part was soaked for 1 h in 20 ml of deionized water at 85 °C. A 5-ml aliquot from the extract solution was loaded into the autosampler for

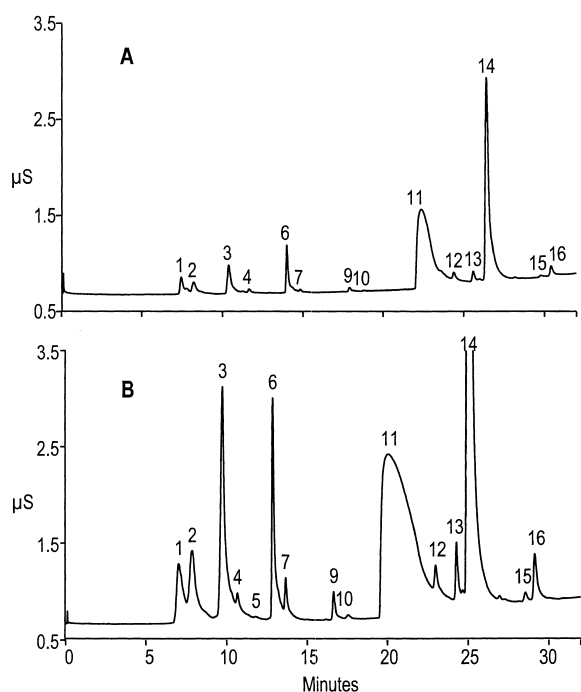


Fig. 6. Analysis of deionized water extract from disk drive clamp. (a) 1-ml Direct injection. Peaks: 1 = fluoride (1.8 µg/l); 2 = acetate (3.1 µg/l); 3 = formate (6.2 µg/l); 4 = acrylate (1.3 µg/l); 6 = chloride (4.3 µg/l); 7 = nitrite (0.10 µg/l); 9 = nitrate (0.70 µg/l); 10 = benzoate (0.36 µg/l); 11 = carbonate; 12 = unidentified; 13 = sulfate (1.5 µg/l); 14 = oxalate (46 µg/l); 15 = phthalate (0.86 µg/l); 16 = phosphate (4.0 µg/l); for chromatographic conditions, see Table 1. (b) 5-ml Preconcentration. Peaks: 1 = fluoride (2.0 µg/l); 2 = acetate (7.7 µg/l); 3 = formate (13 µg/l); 4 = acrylate (1.0 µg/l); 5 = methacrylate (0.065 µg/l); 6 = chloride (5.4 µg/l); 7 = nitrite (1.0 µg/l); 9 = nitrate (0.90 µg/l); 10 = benzoate (0.60 µg/l); 11 = carbonate; 12 = unidentified; 13 = sulfate (1.7 µg/l); 14 = oxalate (46 µg/l); 15 = phthalate (1.0 µg/l); 16 = phosphate (5.0 µg/l); for chromatographic conditions, see Table 2.

IC analysis. Fig. 5 shows representative chromatograms for the analysis of the disk drive spacer extract by both methods. The reported concentration values have not been corrected for the method blank. The anions of interest were detected below 10 µg/l. Two peaks are labeled as ‘unidentified’.

An extract of a disk clamp was also analyzed as shown in Fig. 6. This component is fabricated from 300 series stainless steel and is used to secure the disk to the spindle. Higher levels for the anions were detected in the extract solution of the clamp than from the spacer, especially oxalate which was measured at 46 µg/L. The source of this contaminant is the oxalic acid used for cleaning this part.

Extracts from disk drive parts from the same production lot were analyzed by the 5-ml preconcentration method. Results are presented in Table 3 for the anion analysis of seven blanks and the extracts from five disk spacers and 14 disk clamps. RSDs for the contaminant anions in both the blank and extracts ranged from 20 to 60%. This level of precision is a measure of the variation in component contamination within a production lot. It also reflects the difference in cleanliness of the extraction vessels and autosampler vials. Comparable results were calculated for the 1-ml method (data not shown).

Calibration curves were obtained using standards prepared in deionized water. At least three replicate injections were used at each concentration level. The

Table 3
Trace anion determination for blanks, spacer extracts, and clamp extracts using the 5-ml preconcentration method

Anion	Blank (µg/l±SD) n = 7	Spacer extract (µg/l±SD) n = 5	Clamp extract (µg/l±SD) n = 14
Fluoride	0.11±0.022	2.4±0.22	2.8±0.74
Acetate	4.2±1.0	5.9±1.0	6.3±1.3
Formate	9.3±1.1	8.6±1.5	13±1.6
Acrylate	Trace<0.12	Not detected	0.97±0.18
Methacrylate	Trace<0.11	Trace<0.11	Trace<0.11
Chloride	0.76±0.22	1.4±0.34	5.6±0.95
Nitrite	1.0±0.28	0.21±0.13	0.73±0.25
Bromide	Not detected	0.27±0.070	Trace<0.043
Nitrate	0.14±0.080	0.26±0.060	1.1±0.45
Benzoate	0.65±0.19	0.61±0.18	0.73±0.22
Sulfate	0.43±0.12	0.27±0.14	2.1±1.2
Oxalate	0.38±0.17	Not detected	44±2.5
Phthalate	Not detected	Not detected	1.3±0.19
Phosphate	Not detected	Not detected	4.0±0.79

Table 4
Calibration results for trace anion determination using the 1-ml method

Anion	Data points	r^2	Slope	Intercept	Dynamic range ($\mu\text{g/l}$)
Fluoride	16	0.9942	33.2±1.46	0.19±0.250	1–10
Acetate	9	0.9928	88.7±9.78	−0.07±0.917	5–20
Formate	9	0.9828	118±13.9	0.104±0.826	6–12
Acrylate	16	0.9985	251±5.7	0.092±0.035	0.3–3
Methacrylate	16	0.9967	258±8.6	0.010±0.054	0.3–3
Chloride	9	0.9937	35.1±2.49	0.222±0.119	0.3–3
Nitrite	14	0.9955	78±3.14	−0.079±0.24	1–10
Bromide	22	0.9984	141±2.60	0.028±0.028	0.1–10
Nitrate	15	0.9986	79.7±1.78	−0.015±0.037	0.3–3
Benzoate	21	0.9959	298±9.16	0.25±0.211	1–15
Sulfate	21	0.9918	90.5±3.96	0.078±0.212	1–10
Oxalate	24	0.9989	71.3±1.03	0.956±0.371	1–60
Phthalate	23	0.9990	194±2.7	0.020±0.104	0.5–15
Phosphate	14	0.9982	129±3.40	1.02±0.24	1–15

dynamic range for each analyte in the calibration curve was set to cover its expected concentration range in the extract samples. To accurately determine the area of a peak at trace levels it was necessary on occasion to manually draw the baselines using the tools in the chromatography software. Results for the anions of interest yielded a linear response with coefficients of determination (r^2) greater than 0.98. Table 4 lists the regression data for the 1-ml direct injection method. Calibration slope precision ranged from 1 to 12% RSD for the analytes of interest.

Comparable results were calculated for the 5-ml preconcentration method (data not shown).

Method detection limits (MDLs) for the target analytes were calculated for both methods using peak heights of standards compared to the height of the noise in a representative 1-min portion of the baseline (Table 5). MDLs for each anion were defined as the detectable concentration of an anion giving a peak three-times higher than the background noise ($S/N=3$). The 5-ml preconcentration method has the most sensitivity but requires an additional

Table 5
Method detection limits (MDLs) for analysis of DI water extracts of disk drive components

Anion	1-ml Direct injection MDL ^a ($\mu\text{g/l}$)	5-ml TAC-LP1 preconcentration MDL ^a ($\mu\text{g/l}$)
Fluoride	0.08	0.024
Acetate	0.16	0.072
Formate	0.17	0.038
Acrylate	0.45	0.12
Methacrylate	0.35	0.11
Chloride	0.05	0.014
Nitrite	0.10	0.031
Bromide	0.16	0.043
Nitrate	0.11	0.028
Benzoate	0.71	0.27
Sulfate	0.13	0.028
Oxalate	0.17	0.035
Phthalate	0.37	0.10
Phosphate	0.28	0.076

^a $S/N=3$.

concentrator column, more sample, and more time to load the sample. Conversely, the 1-ml direct injection method has good sensitivity without the need for a preconcentration column. Also, it is important to remember that the lowest quantifiable analyte concentration is generally three to five times greater than the lowest detectable concentration [16].

4. Conclusion

Microbore ion chromatography was applied to the analysis of aqueous extracts of disk drive parts. The IonPac AS17 column with a potassium hydroxide gradient was able to separate fluoride, acetate, formate, acrylate, methacrylate, chloride, nitrite, bromide, nitrate, benzoate, sulfate, oxalate, phthalate, and phosphate. On-line generation of potassium hydroxide eluent allowed for the best sensitivity and method reproducibility. Two methods were evaluated to achieve sensitivity to less than 1 $\mu\text{g}/\text{l}$: 1-ml direct injection and 5-ml preconcentration. The direct injection method had the most efficient peaks with acceptable sensitivity. The preconcentration method had increased sensitivity but less efficient peaks. Both techniques were useful for profiling the anion contaminants in the aqueous extracts of a disk drive spacer and a disk drive clamp.

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