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Application News

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Prominence HIC-20A / Anions / PCB Electronics

Quantitative Analysis of Extractable Inorganic Anions and Succinate in PCB Extract Water by Ion Chromatography

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❑ Introduction

Ionic cleanliness of electronic devices is of importance to the quality and durability of printed circuit board and assembly (PCBA) products. This is because ionic contamination may lead to electronic degradation and failure of PCBA by means of corrosions. Apart from major inorganic anions available in the environment such as chloride and bromide, weak organic anions (WOA) are also found on surface of PCBA due to their uses as activators in solder fluxes in manufacturing [1]. The IPC-TM-650 standard method 2.3.28A [2] lists seven inorganic anions and eight weak organic acids including succinate, and describes the sample extraction procedure using hot water bath at 80°C as well as analysis requirements such as detection of 50 ppb or better using ion chromatography (IC). In this application news, a high sensitivity IC method for quantification of seven inorganic anions and succinate in PCB extract water samples on HIC-20A ion chromatography with suppressed conductivity detector.

❑ Experimental

Analytical conditions

A Shimadzu Prominence™ HIC-20A ion chromatography system with CDD-10Asp conductivity detector and anion suppresser cartridge was employed in this work. A pair of suppresser cartridges were used alternately between analysis status and regeneration. The details of IC analytical conditions are compiled into Table 1.

Table 1: Analytical conditions of anions on Prominence HIC-20A

Column	Shim-pack™ IC-SA3 (250 mmL x 4.0 mm id)
Mobile Phase	3.6 mM Na_2CO_3
Elution Program	Isocratic elution
Flow Rate	1.0 mL/min
Oven Temp.	45°C
Injection	500 µL (full loop)

Preparation of standards and samples

Eight individual anion standards (1000 ppm in aqueous solution) were obtained from Sigma Aldrich and BDH, VWR. A mixed standard was then prepared from the individual stock solutions and diluted serially using Milli-Q® deionized water (>18 MΩ-cm resistivity) to various concentrations as the calibrants. Two PCB extract water samples obtained from a manufacturer were tested as example. These samples were filtered through a 0.22 µm nylon filter before injection to IC system for analysis.

❑ Results and Discussion

IC chromatogram and calibration curves

Figure 1 shows two selected ion chromatograms of mixed standards including seven inorganic anions (F, Cl, NO_2, Br, NO_3, PO_4 and SO_4) and an organic anion succinate (succinic acid). The total run time is 25 minutes with succinate anion eluted as the last peak at 20.7 minutes.

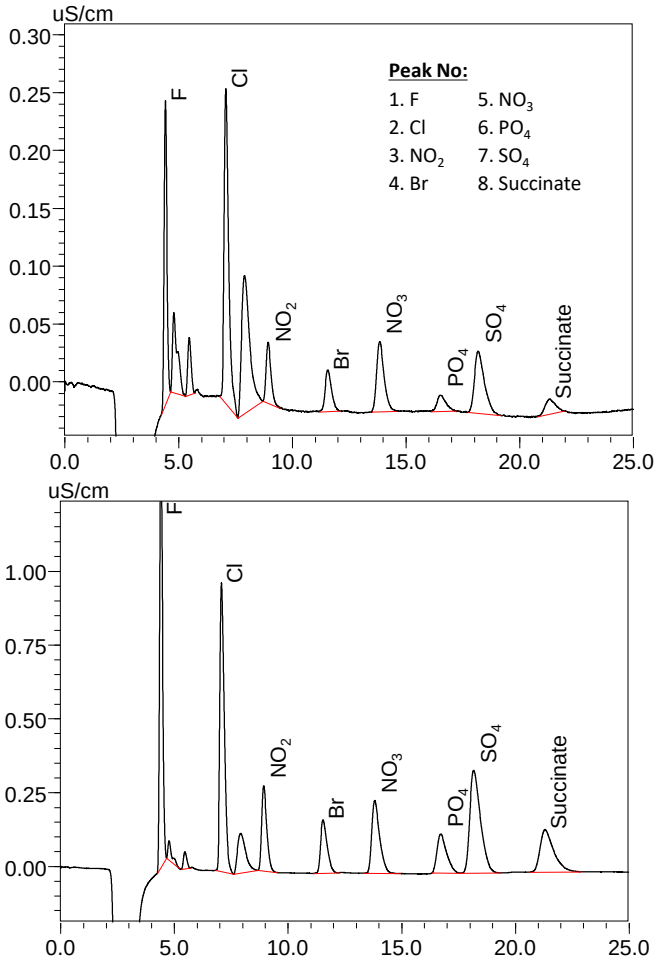

Figure 1: IC chromatograms of mixed anion standards. Concentration (top): 10 ppb for No. 1~4, 5, 7; 20 ppb for No. 6 and 50 ppb for No. 8. (Bottom): 50 ppb for No. 1~4, 5, 7; 100 ppb for No. 6 and 250 ppb for No. 8.

Nine-point external standard calibration curves were established. Four selected calibration curves are displayed in Figure 2. The details of calibration parameters and method performance are

Table 2: Calibration curve parameters and performance of IC method for quantitative determination of eight anions on HIC-20A with suppressed CDD

Peak No.	Anion	RT (min)	Conc. Range (ppb)	R^2	S/N (Lowest Level)	LOD (ppb)	LOQ (ppb)	RSD % (area, n=3)		
								L2 (20 ppb)	L6 (500 ppb)	L8 (2000 ppb)
1	F	4.5	10 ~ 5,000	0.999	219	0.14[1]	0.43[1]	3.9	3.5	1.9
2	Cl	7.1	10 ~ 5,000	0.999	227	0.16[1]	0.50[1]	1.4	3.4	2.4
3	NO_2	8.9	10 ~ 5,000	0.999	43.5	0.7	2.2	3.2	3.1	2.1
4	Br	11.3	10 ~ 5,000	0.998	30.0	1.1	3.2	2.9	3.1	2.3
5	NO_3	13.4	10 ~ 5,000	0.998	50.6	0.7	2.1	1.7	3.3	2.3
6	PO_4	16.4	20 ~ 10,000	0.997	11.6	6.1	18.4	10.1[2]	2.5[2]	2.3[2]
7	SO_4	18.0	10 ~ 5,000	0.999	44.7	0.9	2.8	2.7	3.5	2.4
8	Succinate	20.7	50 ~ 25,000	0.999	10.8	15.0	45.4	8.9[2]	3.7[2]	2.3[2]

[1] *Estimated values for reference only due to S/N > 200;* [2] *Conc. of PO_4 and succinate are 2 and 5 times higher than other anions, respectively.*

shown in Table 2. The calibration ranges are at 10~5000 ppb for all inorganic anions except PO_4 (20~10,000 ppb). For succinate, the calibration curve ranges from 50 ppb to 25,000 ppb. All these calibration curves exhibit good linearity of $R^2 \geq 0.997$ (Table 2). The lowest concentration levels (L1) were used to determine sensitivities of the method based on S/N ratios of 3 and 10 for LOD and LOQ, respectively. The results of the sensitivity evaluation indicate that the current method using Shimadzu anion suppresser CDD detector is sufficient for detection of anions on the surface of PCB and assemblies following IPC-TM-650 method 2.3.28A [2]. Repeatability test was conducted with L2 (20 ppb), L6 (500 ppb) and L8 (2000 ppb). The RSD% obtained ranges at 1.4~10.1% as shown in Table 2.

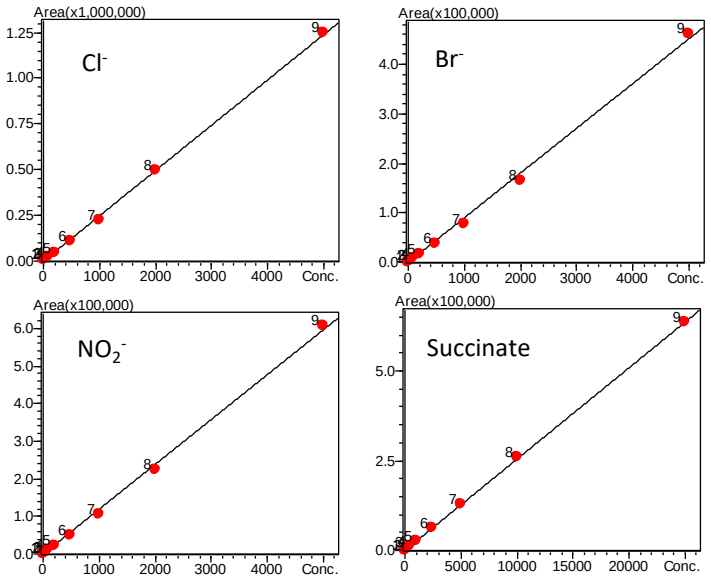

Figure 2: Selected calibration curves of anion standards on HIC-20A.

Application examples

Two PCB extract water samples obtained were analysed using the method established. The IC results are displayed in Figure 3 and the quantitative results are summarized in Table 3. The main anions found in both samples are chloride, nitrate, sulphate and succinate.

❑ Conclusions

An IC method was established for quantitative analysis of eight anions (F, Cl, NO_2, Br, NO_3, PO_4, SO_4 and succinate) in PCB extract water on HIC-20A. The sensitivities for these anions are sufficient as per the requirement in IPC-TM-650 method 2.3.28A.

Disclaimer: The data and instruments presented in this Application News are intended for Research Use Only (RUO). Not for use in diagnostic procedures.

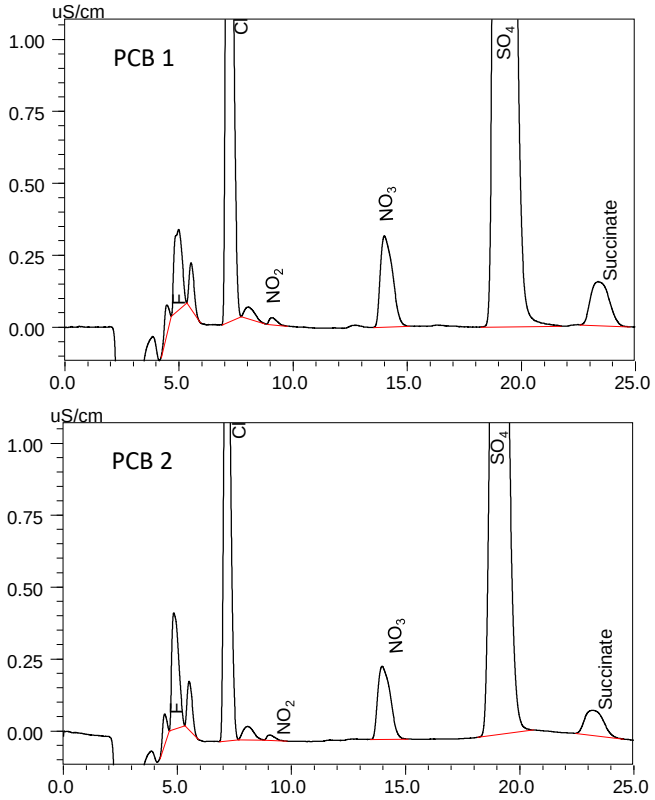

Figure 3: IC Analysis of two PCB extract water samples on HIC-20A.

Table 3: Quantitative results on anions in PCB extract water samples

No.	Anion	Concentration (ppb)	
		PCB 1	PCB 2
1	F	5.6	5.4
2	Cl	273.8	157.8
3	NO_2	7.0	5.8
4	Br	N.D	N.D
5	NO_3	104.4	85.3
6	PO_4	N.D	N.D
7	SO_4	2132.5	831.9
8	Succinate	377.6	231.7

❑ References

1. Jellesen, M. S., Verdingovas, V., Conseil, H., Piotrowska, K., & Ambat, R. (2014). *Corrosion in electronics: Overview of failures and countermeasures.* In Proceedings of EuroCorr 2014
2. Association Connecting Electronics Industries; IPC-TM-650 Test Methods Manual, Method 2.3.28A, https://www.ipc.org/TM/2-3_2-3-28b.pdf

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