

Detection of multiple ions in circuit board

Introduction:

The density of the electrical components on the PCB circuit board is high, and the residual separation on the surface will cause the possibility of separation migration, resulting in open circuit, short circuit and other phenomena. If there is acid residue on the surface of the circuit board, it will corrode the circuit board and reduce the service life of the product.

Detection items (Table 1):

| | | | | | | | |
|--------------|-----------------|-----------------|------------------------------|-----------------|------------------------------|-------------------------------|-------------------------------|
| Anion | F ⁻ | Cl ⁻ | NO ₂ ⁻ | Br ⁻ | NO ₃ ⁻ | SO ₄ ²⁻ | PO ₄ ³⁻ |
| Cation | Li ⁺ | Na ⁺ | NH ₄ ⁺ | K ⁺ | Mg ²⁺ | Ca ²⁺ | |
| Organic acid | Acetic acid | Formic acid | Methane sulfonic acid | Malic acid | Succinic acid | Adipic acid | Phthalic acid |

Keywords: PCB, circuit board, Ion chromatography

Instruments and equipment

- **Ion chromatograph:** CIC-D180
Qingdao Shenghan Chromatography Technology Co., Ltd
- **Ultra pure water machine:** UPT-I-20L
Sichuan youpu Chaochun Technology Co., Ltd



Requirements

Reagents

All reagents used are superior grade pure or better, Purchase certified standard solutions F^- , Cl^- , NO_2^- , Br^- , NO_3^- , SO_4^{2-} , PO_4^{3-} , Li^+ , Na^+ , NH_4^+ , K^+ , Mg^{2+} , Ca^{2+} , acetic acid, formic acid, methanesulfonic acid, malic acid, succinic acid, adipic acid, phthalic acid, standard solutions (1000 mg / L).

Deionized Water

When preparing standard samples manually or diluting real samples, please use ASTM filtration and deionization requirements that meet the specifications listed in the table 2.

Table 2: Deionized water specification.

| Specification | |
|------------------------------|-------------------------------|
| Ions Resistivity | $\geq 18.25 M\Omega \cdot cm$ |
| Organics-TOC | <10ppb |
| Iron/Transition Metals | <1ppb |
| Pyrogens | <0.03Eu/mL |
| Particulates (>0.2 μm) | <1unit/mL |
| Colloids-Silica | <10ppb |
| Bacteria | <1cfu/mL |

Sample preparation

(1) Put the circuit board into a PE self sealing bag, add 640 ml of isopropanol water mixed solution (the volume ratio of isopropanol to water is 75:25), remove the gas, make the extraction solution fully contact with the circuit board, and take a water bath at 80 °C for 1 h.

(2) Pour out the leaching solution into a triangular flask and place it in a 90 °C water bath for 2 hours, so that isopropanol volatilizes and overflows. The remaining liquid is naturally cooled to room temperature, transferred to a 250 ml volumetric flask for constant volume, and passed through 0.22 μm filter membrane injection analysis.

(3) Treat the mixed solution of isopropanol and water of the same volume according to the above steps as the solvent blank of the PE self sealing bag.

Chromatographic conditions (Table 3):

| | Column | Eluent | Flow rate | Column temperature | Injection volume | Suppressor current |
|--------------|----------|------------|------------|--------------------|------------------|--------------------|
| Anion | SH-AC-11 | 10 mM KOH | 1.0 mL/min | 55°C | 25 μL | 60 mA |
| Cation | SH-CC-3L | 5.5 mM MSA | 1.0 mL/min | 35°C | 25 μL | 45 mA |
| Organic acid | SH-AC-23 | 13 mM KOH | 1.0 mL/min | 35°C | 25 μL | 60 mA |

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Serial number:007

Standard chromatogram (Anion)

Standard chromatogram, As shown in below:

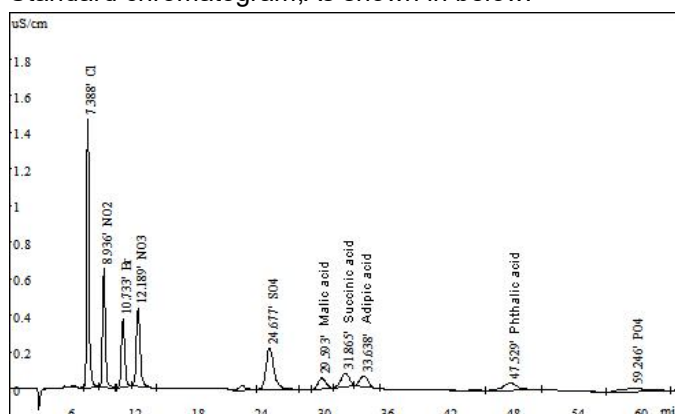


Figure 1. Chromatogram of anion standard sample.

Table 4: Data of anion standard solution

| No. | Reten.time | Name | Concentration | Area | Peak height |
|-----|------------|-------------------------------|---------------|----------|-------------|
| 1 | 7.388 | Cl ⁻ | 1 | 18568621 | 1464610 |
| 2 | 8.936 | NO ₂ ⁻ | 1 | 9875120 | 649078 |
| 3 | 10.733 | Br ⁻ | 1 | 7231764 | 368324 |
| 4 | 12.189 | NO ₃ ⁻ | 1 | 9856235 | 426541 |
| 5 | 24.677 | SO ₄ ²⁻ | 1 | 11953115 | 229956 |
| 6 | 29.593 | Malic acid | 1 | 3054093 | 59917 |
| 7 | 31.865 | Succinic acid | 1 | 3635839 | 71354 |
| 8 | 33.638 | Adipic acid | 1 | 3066828 | 57106 |
| 9 | 47.529 | Phthalic acid | 1 | 4790150 | 45422 |
| 10 | 59.246 | PO ₄ ³⁻ | 1 | 2639442 | 15676 |

Comparison testing (Anion blank)

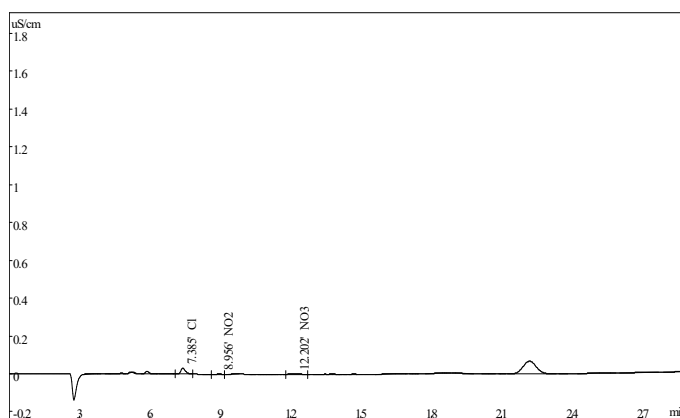


Figure 2. Chromatogram of Anion blank.

Table 5: Data of anion blank

| No. | Reten.time | Name | Concentration | Area | Peak height |
|-----|------------|------------------------------|---------------|--------|-------------|
| 1 | 7.385 | Cl ⁻ | 0.02769 | 514247 | 34878 |
| 2 | 8.956 | NO ₂ ⁻ | 0.004241 | 41876 | 3123 |
| 3 | 12.202 | NO ₃ ⁻ | 0.01434 | 141377 | 6315 |

Sample chromatogram (Anion)

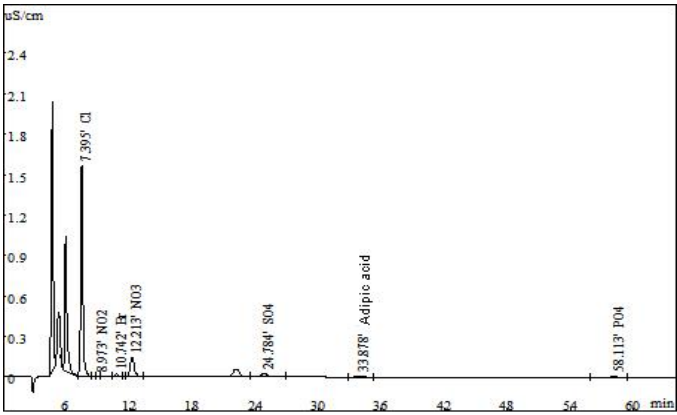


Figure 3. Chromatogram of anion in sample

Standard chromatogram (Cation)

Standard chromatogram,As shown in below:

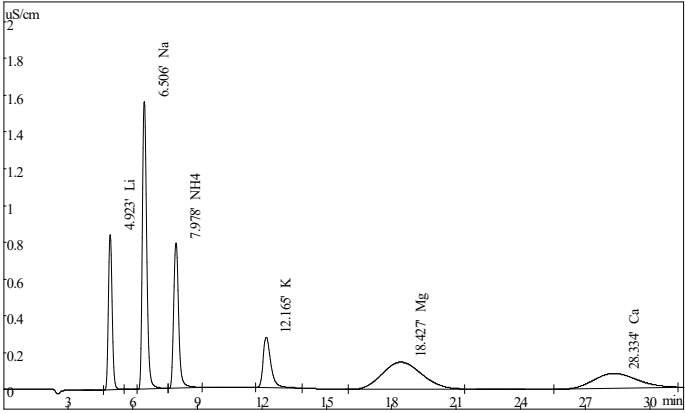


Figure 4. Chromatogram of cation standard sample.

Table 6: Data of cation standard solution

| No. | Reten.time | Name | Concentration | Area | Peak height |
|-----|------------|------------------------------|---------------|----------|-------------|
| 1 | 4.923 | Li ⁺ | 0.1 | 9511119 | 843894 |
| 2 | 6.506 | Na ⁺ | 1 | 20511784 | 1566395 |
| 3 | 7.978 | NH ₄ ⁺ | 0.5 | 11945453 | 789479 |
| 4 | 12.165 | K ⁺ | 0.5 | 6492749 | 272982 |
| 5 | 18.427 | Mg ²⁺ | 0.5 | 18784915 | 147791 |
| 6 | 28.334 | Ca ²⁺ | 0.5 | 12277267 | 83117 |

Comparison testing (Cation blank)

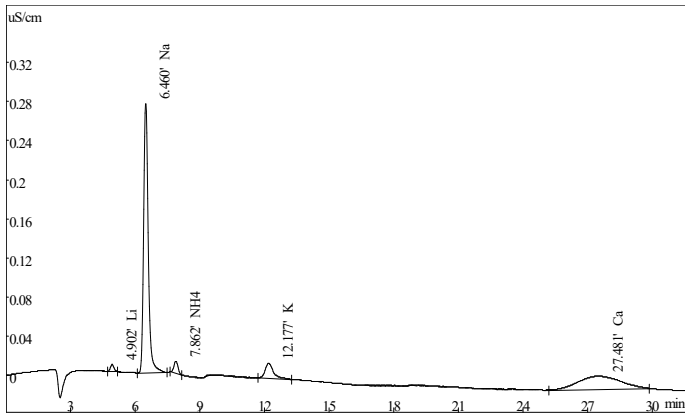


Figure 5. Chromatogram of cation blank.

Table 7: Data of cation blank

| No. | Reten.time | Name | Concentration | Area | Peak height |
|-----|------------|------------------------------|---------------|---------|-------------|
| 1 | 4.902 | Li ⁺ | 0.0008044 | 76504 | 7000 |
| 2 | 6.460 | Na ⁺ | 0.1866 | 3826694 | 275023 |
| 3 | 7.862 | NH ₄ ⁺ | 0.006448 | 154045 | 12199 |
| 4 | 12.177 | K ⁺ | 0.03249 | 421938 | 15979 |
| 5 | 27.481 | Ca ²⁺ | 0.07463 | 1832514 | 13531 |

Sample chromatogram (Cation)

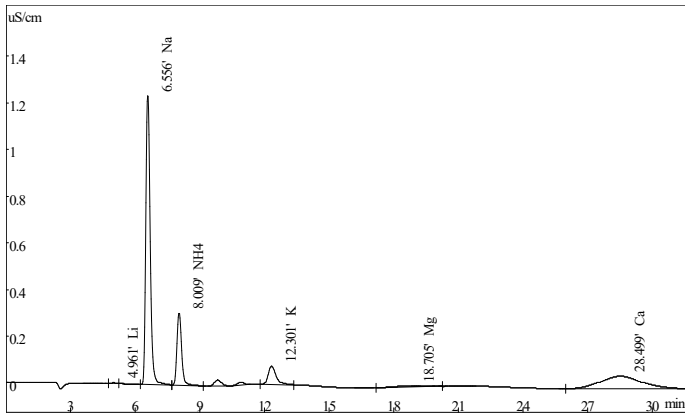


Figure 6. Chromatogram of cation in sample.

Standard chromatogram(Organic acid)

Standard chromatogram,As shown in below:

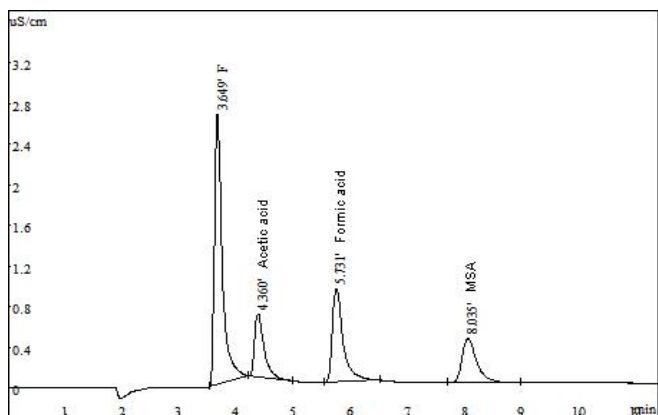


Figure 7. Chromatogram of organic acid standard sample.

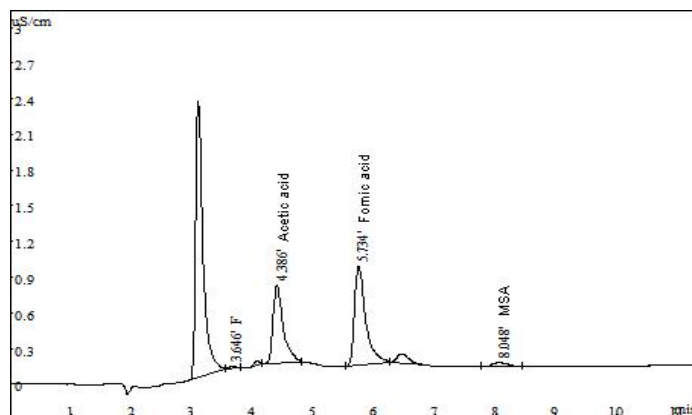


Figure 9. Chromatogram of organic acid in sample

Table 8: Data of organic acid standard solution

| No. | Reten.time | Name | Concentration | Area | Peak height |
|-----|------------|-------------|---------------|----------|-------------|
| 1 | 3.649 | F | 1 | 24251170 | 2652791 |
| 2 | 4.360 | Acetic acid | 1 | 6866639 | 624090 |
| 3 | 5.731 | Formic acid | 1 | 11723651 | 923365 |
| 4 | 8.035 | MSA | 1.239 | 7429014 | 429109 |

Comparison testing (Organic acid blank)

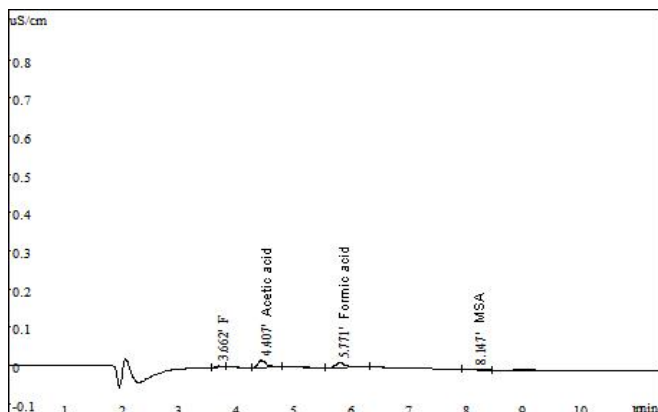


Figure 8. Chromatogram of organic acid blank.

Table 9: Data of organic acid blank

| No. | Reten.time | Name | Concentration | Area | Peak height |
|-----|------------|----------------|---------------|--------|-------------|
| 1 | 3.662 | F ⁻ | 0.001355 | 32871 | 4566 |
| 2 | 4.407 | Acetic acid | 0.03596 | 246933 | 21908 |
| 3 | 5.771 | Formic acid | 0.01702 | 199494 | 14975 |
| 4 | 8.147 | MSA | 0.007238 | 43401 | 2512 |

Sample chromatogram (Organic acid)

Results and calculations

Table 10: Sample test result

| Anion | Concentration (µg/cm ²) | | | | | | |
|---------------|-------------------------------------|-----------------|------------------------------|-----------------|------------------------------|-------------------------------|-------------------------------|
| | F ⁻ | Cl ⁻ | NO ₂ ⁻ | Br ⁻ | NO ₃ ⁻ | SO ₄ ²⁻ | PO ₄ ³⁻ |
| Concentration | 0.0007859 | 0.2035 | ND | 0.01177 | 0.07052 | 0.02375 | 0.06358 |
| Cation | Li ⁺ | Na ⁺ | NH ₄ ⁺ | K ⁺ | Mg ²⁺ | Ca ²⁺ | |
| Concentration | ND | 0.1227 | 0.03614 | 0.02491 | 0.002893 | 0.04727 | |
| Organic acid | Acetic acid | Formic acid | MSA | Malic acid | Succinic acid | Adipic acid | Phthalic acid |
| Concentration | 0.2047 | 0.1582 | 0.01261 | ND | ND | 0.05501 | ND |

Remarks:

- (1) Nd means no detection or lower than the detection limit, or lower than the control group;
- (2) Ion content on circuit board surface (µ G / cm²) = concentration of extraction solution (mg / L) * 250 ml / surface area of circuit board (cm²), and the surface area of circuit board is calculated as 1255.6 cm²;
- (3) Because there are various components on the circuit board with different size bumps, the extraction efficiency of isopropanol water solvent may be affected. The results are for reference only;
- (4) The test results of different methods and laboratories will be different.

Precautions

As the organic acid sample is easy to deteriorate, the organic acid mixed standard in this experiment should be used and prepared now. The circuit board extract should be injected for analysis as soon as possible and stored at 4 °C for refrigeration if necessary; It is easy to be polluted during the experiment, and the experimental personnel are required to operate in strict accordance with the operating procedures.

Feasibility analysis and conclusion

Through the above experiments, it is proved that the detection method has good separation and is suitable for

the determination of the content of the components to be measured in the sample.