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Application News

No. L587

High Performance Liquid Chromatograph Nexera™ X3

# High-speed Simultaneous Analysis of Passivators and Furanic Compounds in Insulating Liquid

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## User Benefits

- Time and labor for switching of analyses can be greatly reduced by unifying two analytical methods into one approach.
- Labor and consumable cost for pretreatment can be greatly reduced because the pretreatment of the passivators and furanic compounds can be conducted at the same time.

## ■ Introduction

Application News No. L576 introduced the simultaneous analysis of passivators and furanic compounds in an insulating liquid.

Benzotriazole (BTA) and tolutriazole derivatives (TTAA) are added into insulating liquids as a passivator, the analytical method of which is specified by British Standards (BS 148:2009). Furanic compounds are used as an indicator of degradation in electrical equipment, the analytical method of which is specified by ASTM D5837-15.

In general, although such analyses are conducted individually, the analytical method introduced in Application News No. L576 enables the simultaneous analysis of these compounds. Because this analytical method was designed for a conventional HPLC system, 30 min is required for one analysis. This article introduces a faster method using an ultra high performance liquid chromatograph Nexera X3.

## ■ Analysis of Standard Solution

Fig. 1 shows the chemical structures of passivators and furanic compounds. The standard solutions were prepared following ASTM. BTA, TTAA, and five furanic compounds were weighed, dissolved in acetonitrile, and diluted with water. In this analysis, Irgamet® 39* (manufactured by BASF) was used as a standard of TTAA because the standard of TTAA is not commercially available.

Table 1 and Fig. 2 show the analytical conditions and chromatogram of the standard solution, respectively. The sufficient separation of seven compounds was achieved in only 1 min in this analysis, although 10 min is required in Application News No. L576. Because the TTAA is a mixture of several isomers and their peaks are occasionally separated, an analytical condition is adjusted to detect the TTAA as one peak.

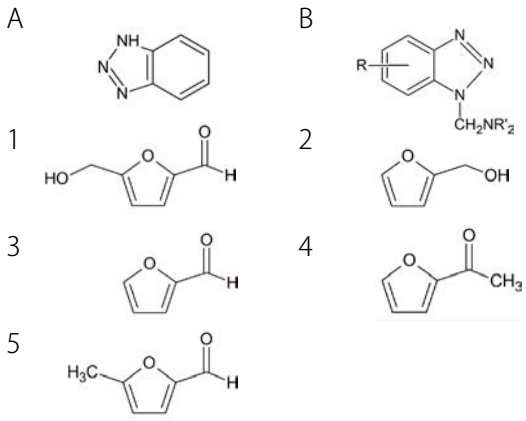


Fig. 1 Chemical Structures
Passivators (A: BTA, B: TTAA)
Furanic Compounds (1: 5-hydroxylmethyl-2-furaldehyde (5HMF), 2: furfuryl alcohol (2FOL), 3: 2-furaldehyde (2FAL), 4: 2-acetylfuran (2ACF), 5: 5-methyl-2-furaldehyde (5MEF))

Table 1 Analytical Conditions

| | | |
|---|---|---|
| System | : | Nexera X3 |
| Column | : | Shim-pack™ XR-ODS III*1 (75 mm × 2.0 mm I.D., 1.6 µm) |
| Mobile Phase | : | A) Water, B) Acetonitrile |
| Time Program | : | B conc. 20% (0–0.3 min) → 90% (1 min) → 100% (1.01–3 min) → 20% (3.01–5 min) |
| Flow Rate | : | 0.7 mL/min |
| Column Temp. | : | 50 ℃ |
| Injection Vol. | : | 5 µL |
| Vial | : | LabTotal Vial for LC/LCMS (Shimadzu GLC)*2 |
| Detection | : | PDA detector (SPD-M40) at 280, 220, and 260 nm |

*1 P/N: 228-59922-92, *2 P/N: 227-34001-01

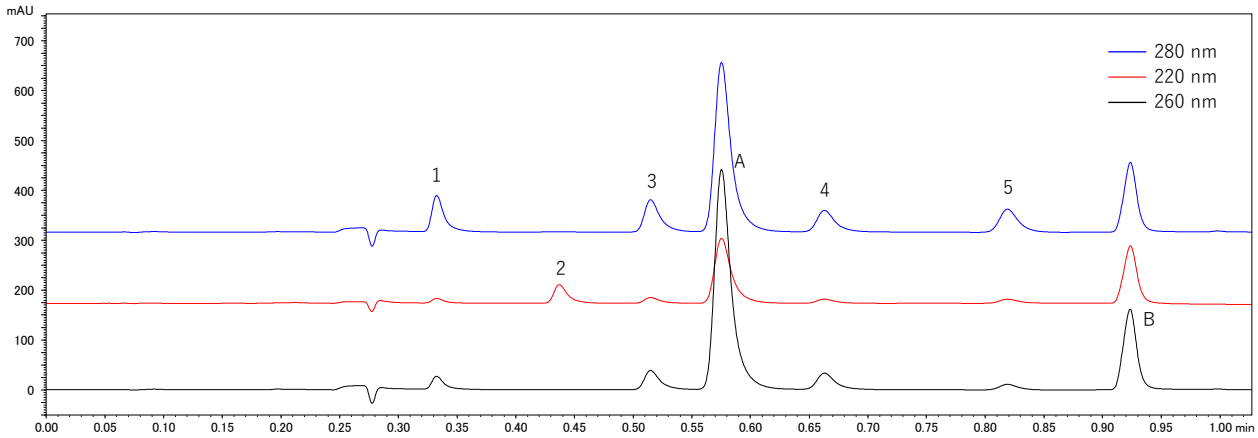


Fig. 2 Chromatogram of Standard Solution
BTA, TTAA: 20000 µg/L each, Furanic Compounds: 1000 µg/L each

## ■ Linearity and Repeatability

Calibration curves for BTA and TTAA were prepared from the chromatograms of standard solutions within the range of 200, 1000, 5000, 10000, and 20000 µg/L, and those for five furanic compounds were prepared in the same manner within the range of 10, 50, 250, 500, and 1000 µg/L. Their linearity ($r^2$) was evaluated, and the area repeatability was also evaluated through the repeated analysis of the standard solution at the highest concentration. Superior linearity and repeatability were obtained for each compound, as shown in Table 2.

Table 2 Detection Wavelength, Linearity, and Repeatability

| | Detection (nm) | Linearity ($r^2$) | Repeatability (%RSD) |
|---|---|---|---|
| BTA (A) | 260 nm | > 0.9999 | 0.12 |
| TTAA (B) | 260 nm | > 0.9999 | 0.19 |
| 5HMF (1) | 280 nm | > 0.9999 | 0.13 |
| 2FOL (2) | 220 nm | > 0.9999 | 0.11 |
| 2FAL (3) | 280 nm | > 0.9999 | 0.24 |
| 2ACF (4) | 280 nm | > 0.9999 | 0.087 |
| 5MEF (5) | 280 nm | > 0.9999 | 0.14 |

## ■ Sample Pretreatment and Recovery Test

The samples were pretreated following ASTM, as shown in Fig. 3. To evaluate the validity of this pretreatment method, a recovery test of each compound was conducted. The BTA, TTAA, and five furanic compounds dissolved in toluene were added into white oil, and their recovery rate was calculated. Table 3 shows the results of the recovery test and Fig. 4 shows its chromatogram. Sufficient recovery and superior reproducibility (n = 3) were obtained for each compound, indicating that this pretreat method is valid.

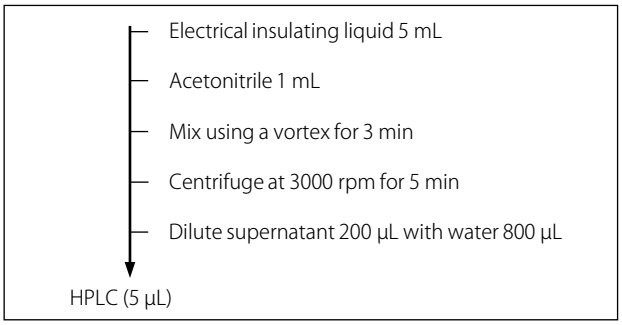


Fig. 3 Protocol of Pretreatment

Table 3 Recovery and Reproducibility (n = 3)

| | Recovery (%) | Reproducibility (%RSD) |
|---|---|---|
| BTA (A) | 89 | 0.85 |
| TTAA (B) | 86 | 0.84 |
| 5HMF (1) | 98 | 0.78 |
| 2FOL (2) | 102 | 0.71 |
| 2FAL (3) | 99 | 1.2 |
| 2ACF (4) | 99 | 0.85 |
| 5MEF (5) | 97 | 1.4 |

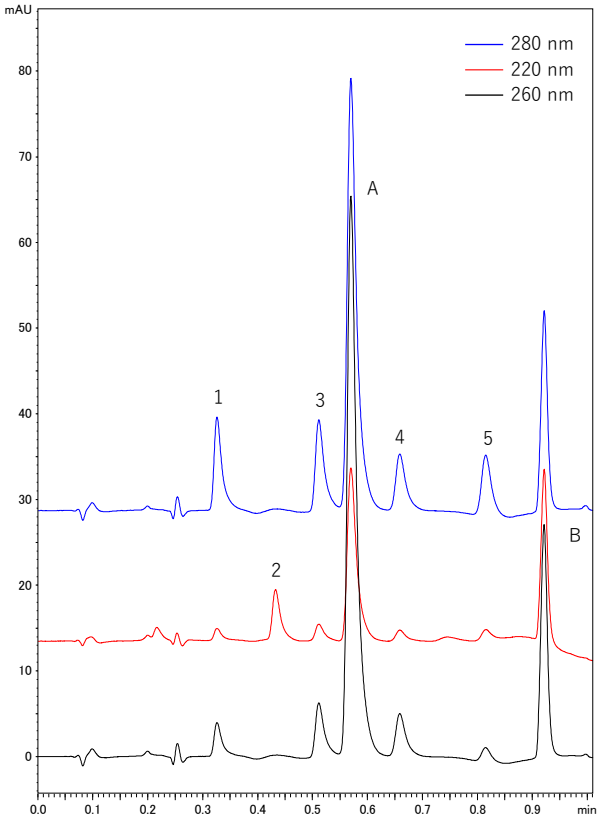


図 4 Chromatogram of Recovery Test
BTA, TTAA: 20000 µg/L each, Furanic Compounds: 1000 µg/L each

## ■ Conclusion

The analytical method described herein enables the simultaneous analysis of passivators and five furanic compounds in only 5 min, although two analyses are generally required. The time and labor required for switching analyses can be greatly reduced using this method. Moreover, labor and cost for pretreatment can be greatly reduced because pretreatment of the passivators and furanic compounds can be conducted at the same time.

*Irgamet® 39 was provided by BASF Japan Ltd.



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First Edition: Dec. 2021