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應用手冊

Monitoring Waterborne Wood Preservatives

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Abstract

This application note describes a method using Waters Breeze 2 HPLC System, and an Atlantis T3 Column to separate five waterborne wood preservative compounds that commonly appear in the open literature on wood treatment.

Introduction

Wood is a common construction material for buildings, furniture, highways, and foot bridges; wetland boardwalks, and other structure in or over water, or in sensitive environments. These wood products are extremely susceptible to naturally occurring wood destroying pestilence, such as termites and fungi. Consequently, wood products need protection to ensure longer life and increase durability. Wood preservatives are chemical protectants and are primarily divided into two classes. The first includes oil-borne preservatives, such as petroleum solutions of pentachlorophenol and creosote. The second class is comprised of the waterborne preservatives. Because waterborne preservatives leave wood surfaces clean, paintable, and free from objectionable odor, this class of preservatives is widely used when cleanliness and paintability of the final treated wood product are required.

Typically, wood preservation treatment is performed on boards, poles, planks, and the like at lumber processing facilities before shipment for construction. Wood treaters rely on compound mixtures of varying cost from wood preservative vendors.[1] If the composition of the preservative solution is incorrect, wood treaters risk releasing boards and planks that could develop mold and decay. This would be extremely costly in terms lost reputation and potential product recalls. To minimize this potential loss, wood treaters monitor concentrations of the organic components in the compound mix. If an HPLC System is not available on-site, samples are sent off-site for analysis, which can take days or weeks. Outsourcing the testing can delay product release, or unnecessarily increase the use of costly preservatives by estimating high additions to treatment tanks to avoid damages.

This application note describes a method using Waters Breeze 2 HPLC System, and an Atlantis T3 Column to separate five waterborne wood preservative compounds that commonly appear in the open literature on wood treatment.[2-3] The analytes are tebuconazole (TEB), propiconazole (PROP), 5- chloro-2-methyl-4-isothiazolin-3-one (CMIT), 2- methyl-4-isothiazolin-3-one (MIT), and 2- octyl-4-isothiazolin-3-one (OCIT), as shown in Figure 1.

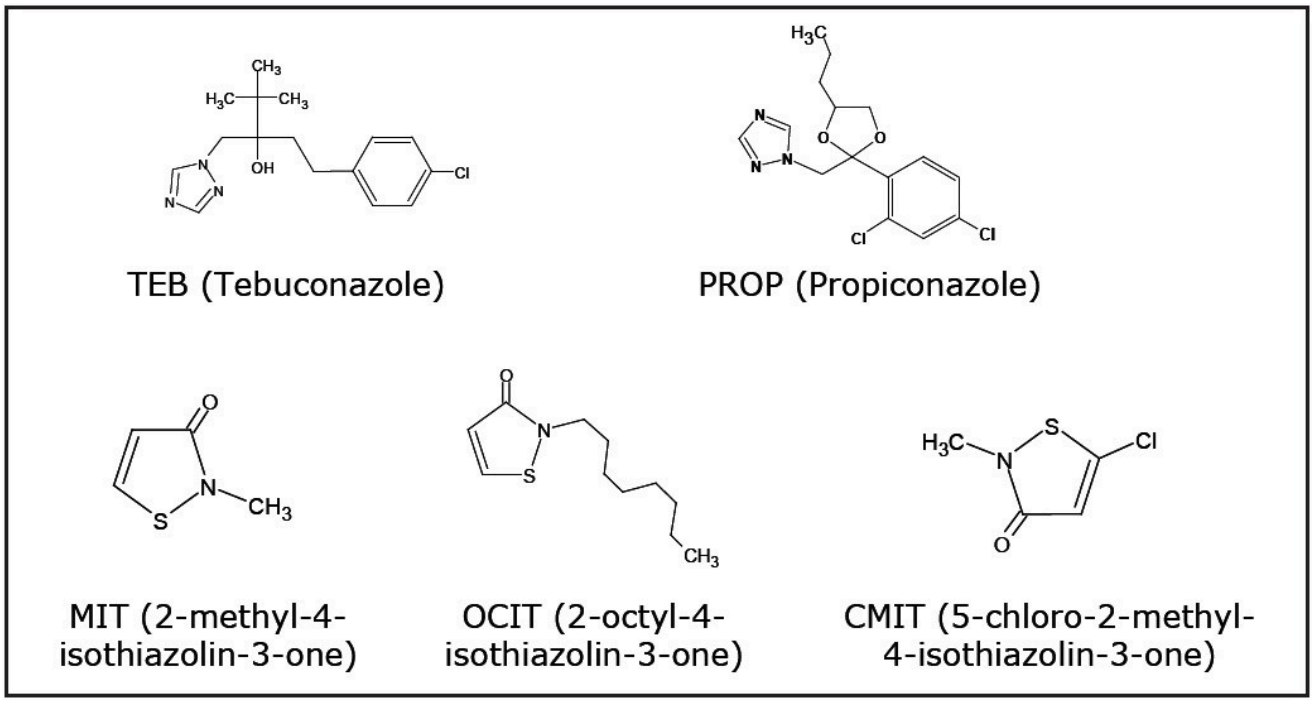

Figure 1. Five common waterborne wood preservative compounds.

Experimental

The Breeze 2 HPLC System used in this method is comprised of the Waters 1525 Binary HPLC Pump, Waters 2707 Autosampler, Waters 2489 UV/Vis Detector, column heater, and Breeze 2 Software.

LC Conditions

System:	Breeze 2 HPLC
Software:	Breeze 2
Column:	Atlantis T3 4.6 x 150 mm 5 µm
Column Temp.:	35 °C
Flow rate:	1.0 mL/min

Injection volume:	10 µL
Needle wash:	80/20 Water/acetonitrile
Mobile phase A:	Water
Mobile phase B:	Acetonitrile

Gradient:

Time (min)	Flow (mL/min)	%A	%B	Curve
0.0	1	80	20	6
7.0	1	30	70	6
15.0	1	15	85	6
15.1	1	80	20	6
25.0	1	80	20	6

Detector Conditions

Wavelengths:	230, 275 nm
Filter time constant:	Fast
Sampling rate:	1 point/s
Data mode:	Absorbance

Autosampler Conditions

Sample loop option:	Partial loop with needle overfill
Overfill volume:	30 µL

Wash solvent:	Water
Wash volume:	500 µL

Materials and Standard Preparation

Tebuconazole, Propiconazole, CMIT, and OCIT were purchased from Sigma-Aldrich. MIT was purchased from Fluka. A 1000 ppm stock solution of MIT was prepared in Methanol (HPLC grade), while 1000 ppm stock solutions of Tebuconazole, Propiconazole, OCIT, and CMIT were prepared in Acetonitrile (HPLC grade). The stock standard mixture solution was prepared by mixing certain amounts of the compounds stock solutions into a 10 mL volumetric flask and diluting up to 10 mL with water (Millipore Milli-Q). Portions of the stock standard solution were diluted with water to make a series of standard solutions and the concentrations are listed in Table 1. The final solutions were filtered with 0.2 µm filters (13 mm Nylon mini spike, WAT 200562).

Std	MIT	CMIT	OCIT	TEB	PROP
1	1.25	62.5	6.25	25	12.5
2	2.5	125	12.5	50	25
3	5	250	25	100	50
4	10	500	50	200	100

Table 1. Concentrations of standards (values are in ppm).

Results and Discussion

Because wood preservatives are proprietary mixtures, only mixtures of standard materials commonly used in the wood industry were used to illustrate the application with a Breeze 2 HPLC System. TEB, PROP, MIT, CMIT, and OCIT are easy to separate, identify, and quantify using an Atlantis T3 Column with gradient elution. All five compounds eluted in less than 12 minutes as shown in Figure 2. Using two UV detection channels instead of one channel provided better quantification, good sensitivity, and accuracy for this combination of five compounds. For maximum sensitivity, the UV detection wavelength for TEB and PROP was 230 nm, whereas, the UV detection wavelength for MIT, CMIT and OCIT, was 275 nm. Figure 2A is a representative chromatogram for TEB and PROP at 230 nm. (Peaks were also evident for MIT, CMIT and OCIT at this wavelength.) Figure 2B is a

representative chromatogram for MIT, CMIT, and OCIT at 275 nm. (Peaks for TEB and PROP were evident).

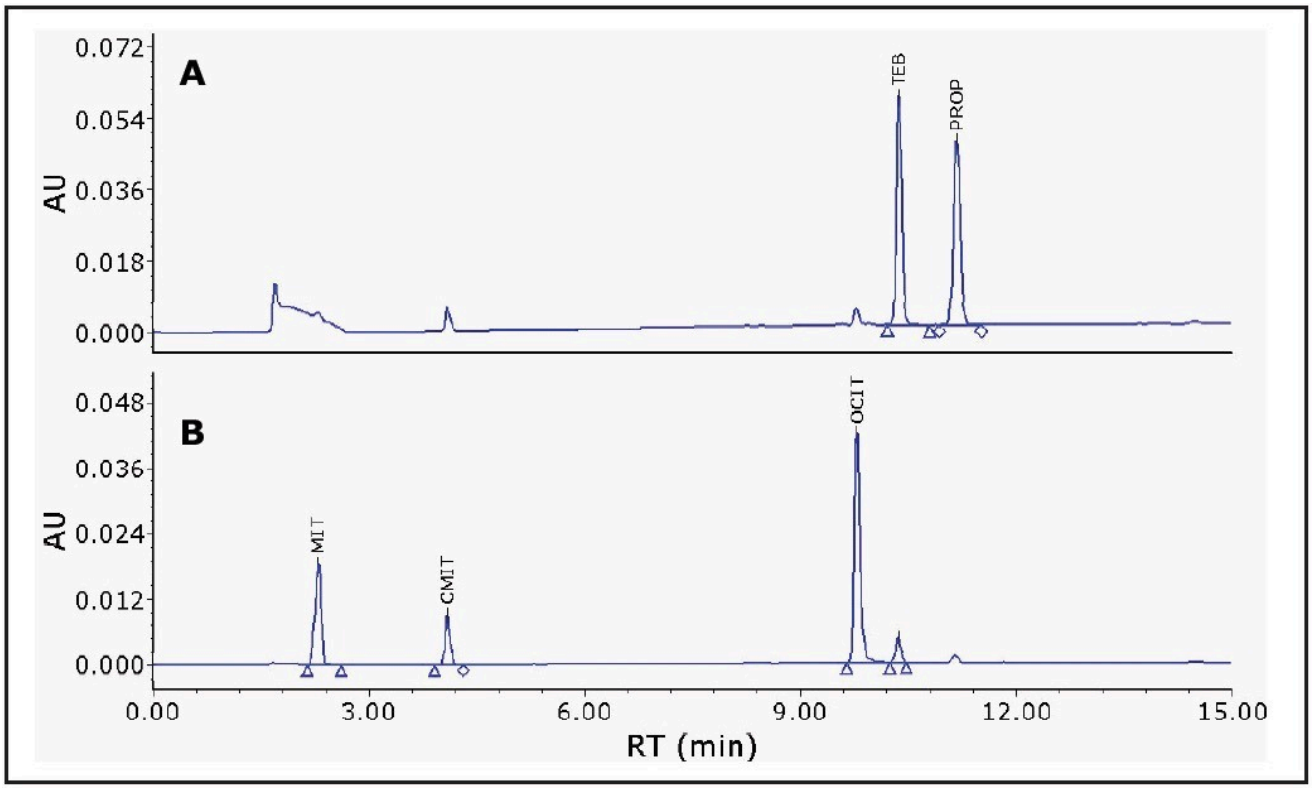

Figure 2. Chromatograms of level 2 wood preservative standard using gradient elution A) TEB and PROP (230 nm); B) MIT, CMIT, and OCIT (275 nm).

Both temperature and column choice are important considerations for this type of analysis. The Atlantis T3 Column provides good retention for the polar compounds, MIT and CMIT. Because CMIT degrades to MIT at elevated temperatures, the temperature was maintained at 35 °C. Figure 3 is an overlay of calibration curves; linearity (R^2) was greater than 0.996 for all five compounds.

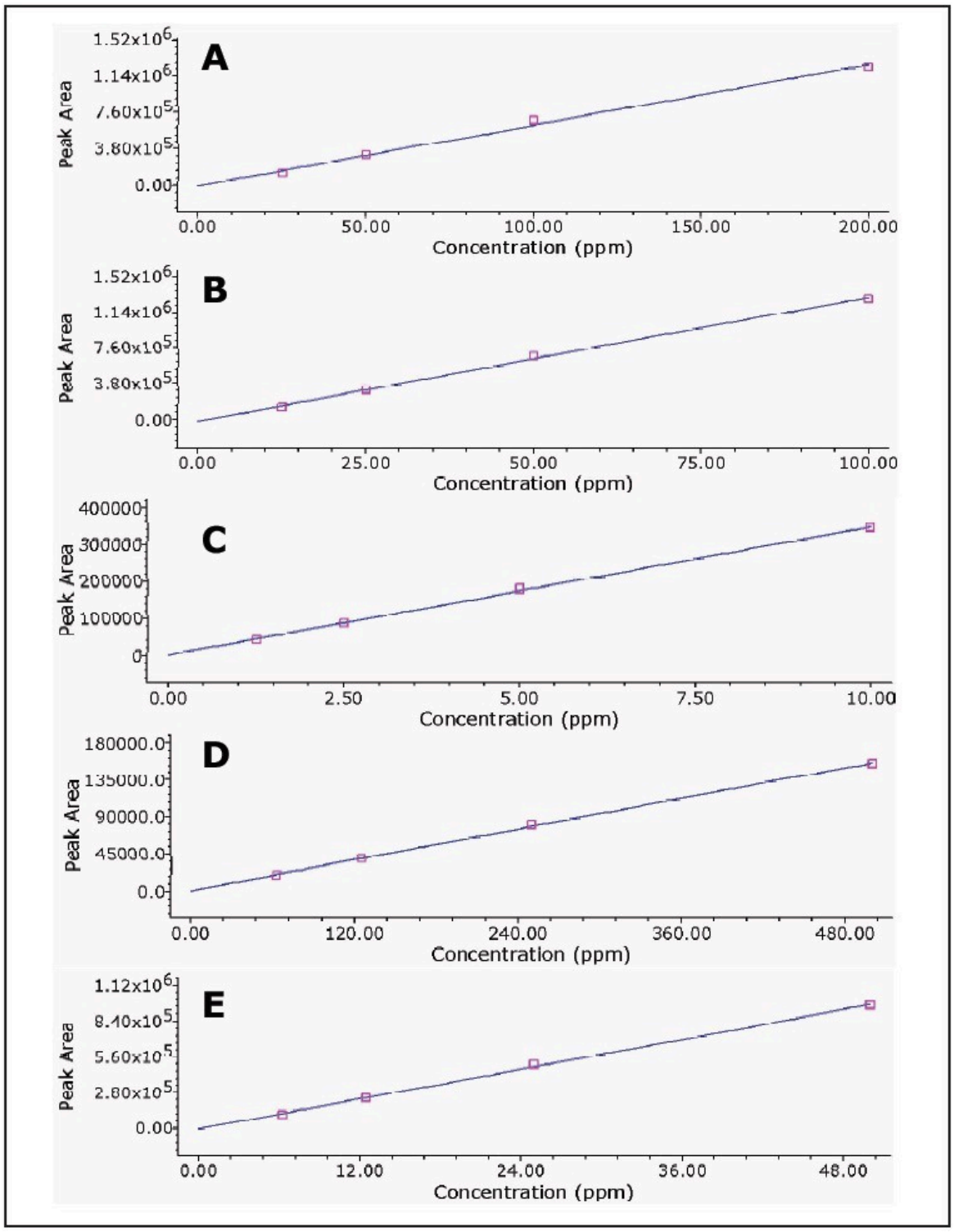

Figure 3. Calibration curves for A) TEB (25 to 200 ppm), B) PROP (12.5 to 100 ppm), C) MIT (1.25 to 10 ppm), D) CMIT (62.5 to 500 ppm), and E) OCIT (6.25 to 50 ppm).

Conclusion

Common waterborne wood preservatives TEB, PROP, MIT, CMIT, and OCIT are easy to analyze using the Breeze 2 HPLC System and Software. In less than 12 minutes, all five compounds eluted from the column. Sample to sample runtime was 25 minutes using gradient elution to ensure that wood related contaminants did not interfere in the analysis. The HPLC methodology described here includes time to flush column contaminants that could be present when sample wood preservative tanks or preserved wood samples are analyzed. Depending upon the commercial mixture of waterborne wood preservatives, there is potential to shorten the method to suit individual needs.

This methodology is easy-to-use, and allows wood treaters to rapidly determine values for organic waterborne wood preservative compound mixtures at the plant treatment site. The method is suitable for commercial formulations, and can provides a number of business benefits, including an increase in productivity, and a reduction in operational costs.

References

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