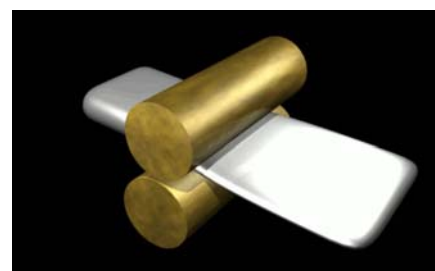


## Application Note

### ► Determination of BHT in rolling oil

Category	Chemical analysis
Matrix	Rolling oil
Method	HPLC
Keywords	BHT, butylated hydroxytoluene, 2,6-di-tert-butyl-4-methylphenol, antioxidants, E321
Analytes	BHT
ID	VCH0003N, 05/10, updated 05/11



From: en.wikipedia.org/wiki/File:Rolling.gif

#### Summary

A robust and sensitive HPLC method for the determination of butylated hydroxytoluene (BHT, 2,6-di-tert-butyl-4-methylphenol) also known as E321 in rolling oil is demonstrated. Furthermore the usage of small column dimensions and particle sizes results in significantly reduced consumption of mobile phase and accelerated analysis. All matrix substances are adequately separated from the main peak making the method very reliable. The fresh base rolling oil sample was shown to contain no BHT additive.

#### Introduction

For the fabrication of sheets and flat products a considerable quantity of rolling oil is needed. This rolling oil avoids undesirable effects caused on extensive friction between strip and roll. Free radicals which will arise from oil components, oxygen and heat lead to a formation of oxidation products in the rolling oil. These oxidation products can result in damages to the final products. To avoid this risk and to extend the life-time of the rolling oil an antioxidant like BHT has to be added.<sup>1</sup> The following figure shows the oxidative degradation of BHT.

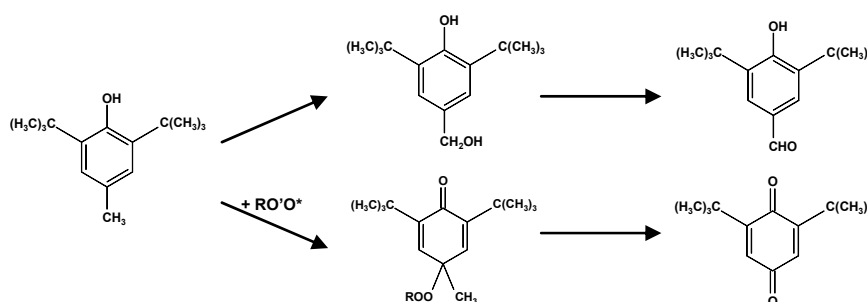


Fig.1

Oxidative degradation of BHT

For an environmentally friendly and cost-effective production, and to achieve consistent product quality, the amount of this antioxidant should be kept constant. This requires frequent monitoring antioxidant levels in the oil emphasizing the need for an easy and robust analysis method.

In this application we will analyse and compare a fresh rolling oil sample without additive and a sample of used rolling oil containing BHT. First we decided to work in normal phase mode due to the solubility of the sample matrix. We selected Si, CN and C<sub>6</sub>H<sub>5</sub> stationary phases for the column screening. The Si phase was selected for the polar interactions, the C<sub>6</sub>H<sub>5</sub> for the pi-pi-interactions and the CN for a mixture of both. The CN column showed the highest selectivity and the best peak shape. For this reason we began method development with this type of column.

### Experimental : Sample preparation

9 mg of fresh rolling oil were diluted in 1 ml of mobile phase and finally filtrated with a 0.45 µm syringe filter.

9 mg of the used rolling oil spiked with BHT were diluted in 1 ml of mobile phase and filtrated with a 0.45 µm syringe filter.

### Experimental : Preparation of standard solution

10 mg of BHT were weighed in a 5 ml volumetric flask, dissolved in approx. 4 ml of mobile phase and filled up to the volumetric mark. The concentrations 0.25 mg/ml, 0.1 mg/ml, 0.01 mg/ml, and 0.001 mg/ml were prepared from stock solution.

### Method parameters

Column	Eurospher 100-5 CN, 250 x 3 mm
Mobile Phase	Heptane/2-butanol 99.9:0.1 (v/v)
Flow rate	0.30 ml/min
Injection volume	10 µl
Column temperature	20°C
System pressure	bar
Detection	UV, 254 nm
Run time	7 min

### Results

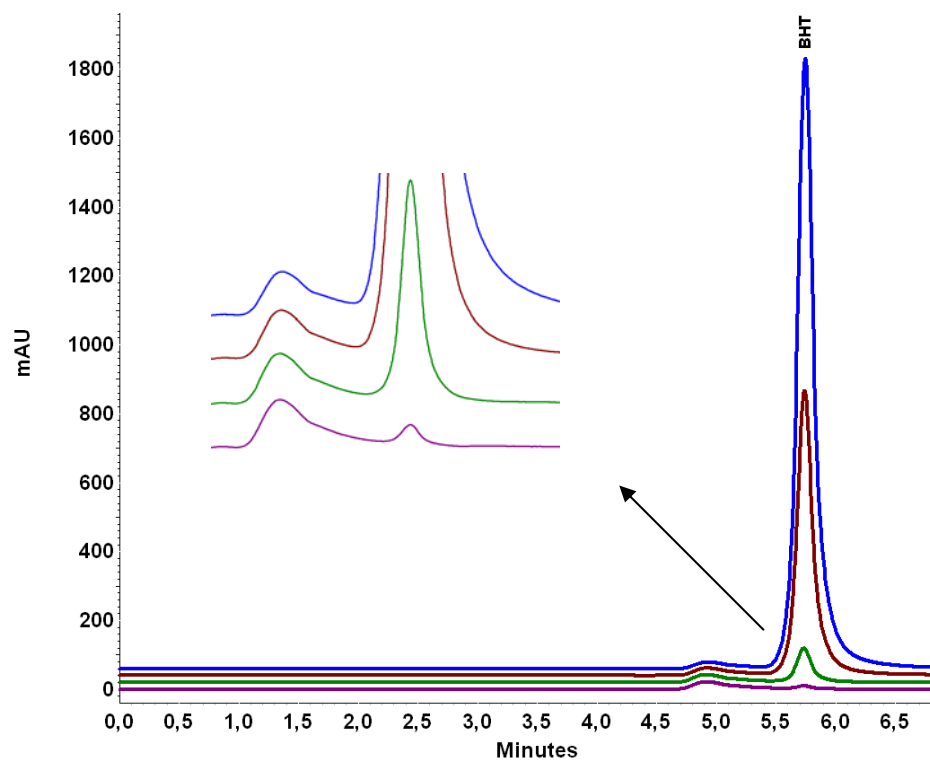


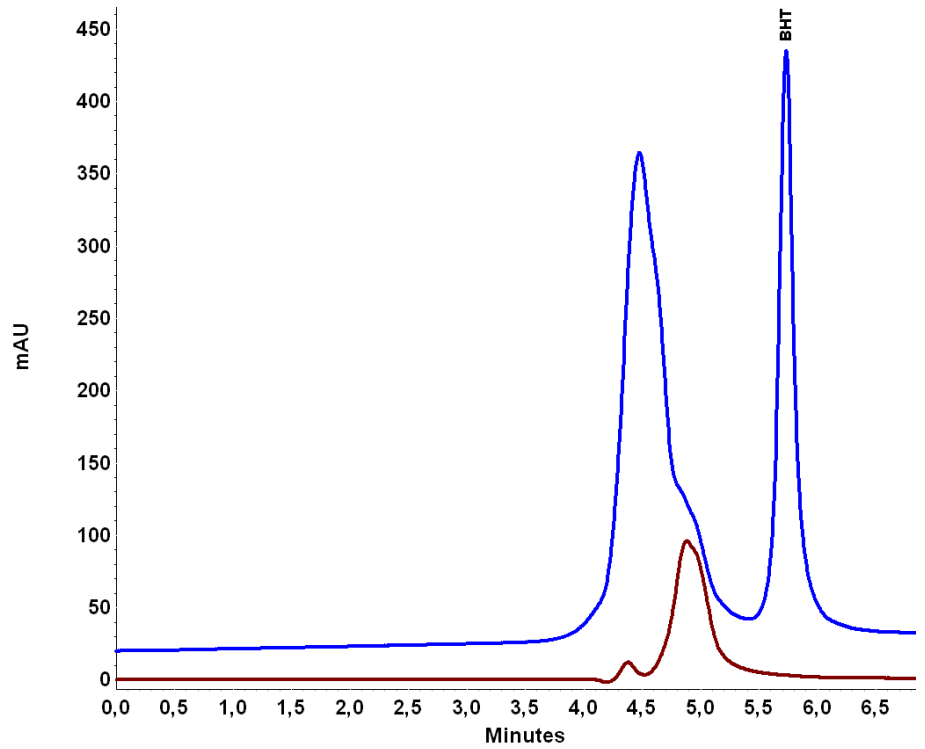
Fig. 2

Overlay of 4 standard solutions of BHT

Substance	Concentration	Peak area (average of three)
BHT (violet)	0.001 mg/ml	77856
BHT (green)	0.01 mg/ml	893002
BHT (red)	0.1 mg/ml	8646995
BHT (blue)	0.25 mg/ml	19702750

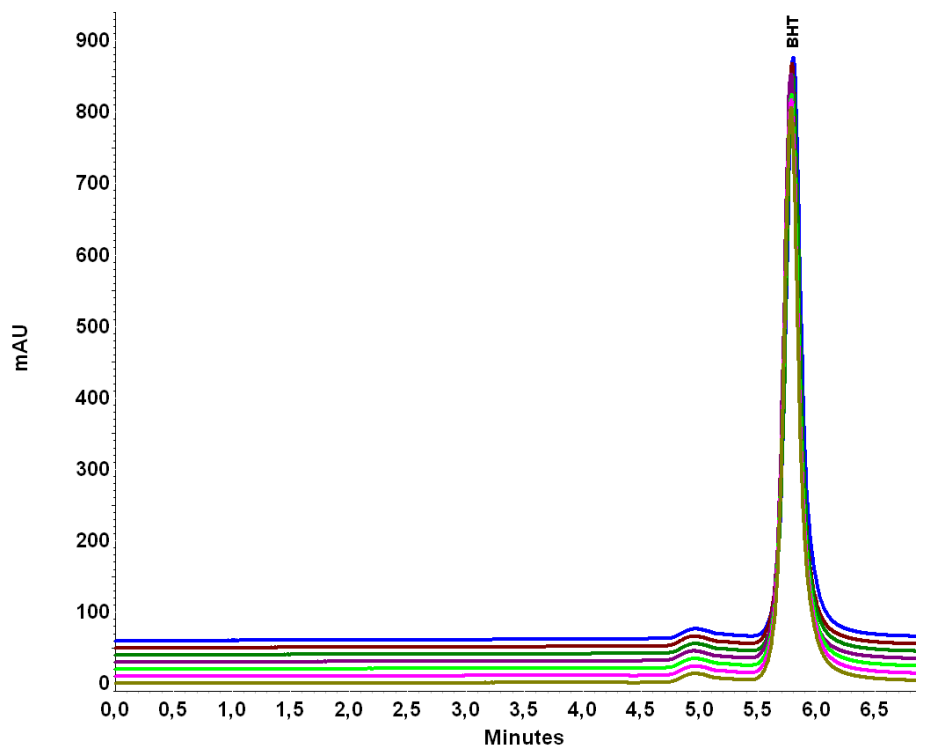
  

Substance	$t_R$	LOD
BHT	5.791 min	0.6 ng



**Fig. 3**  
Overlay of fresh (red) and spiked used rolling oil (blue)

Sample	Peak area (average of two)	Amount BHT per 9.8 mg sample
fresh rolling oil	0	0
spiked rolling oil	3812187	45.6 µg



**Fig. 4**  
Overlay of 7 injections of BHT standard solution

**Method performance**

Limit of detection	ng range (S/N = 3)
Linearity (r <sup>2</sup> )	0.999

<b>Linearity range</b>	10 to 2500 ng BHT
<b>Retention time precision*</b>	< 0.2 % RSD BHT
<b>Peak area precision*</b>	< 1.1 % RSD BHT

\* calculated over 7 replicate runs

## Conclusion

A very reliable and fast separation for the in-process control of rolling oil with good peak symmetry (figures 2-4) is easily accomplished by normal-phase HPLC using the Eurospher CN column and a Smartline HPLC system. Combined with the very easy sample preparation the presented method is a robust and low-cost solution.

Solvent consumption can be further reduced by switching to smaller column dimensions. In this application we saved approximately 57% mobile phase by using a 3 mm ID column instead of a 4.6 mm ID standard sized column.

## References

- 1 R. Fankel, I. Slad, Bestimmung von Butylhydroxytoluol (BHT) in Mischfutter mittels HPLC, Fresenius Z Anal Chem (1988) 331:760

## Physical properties of recommended column



The Eurospher CN packing material can be used in reversed phase, normal phase and polar organic mode and is suitable for substances containing pi-pi double bonds and polar groups. The upper limits regarding temperature and pressure are 50°C and 400 bars for these columns.

<b>Stationary phase</b>	Eurospher 100-5 CN
<b>USP code</b>	L10
<b>Particle size</b>	5 µm
<b>Form</b>	spherical
<b>Dimensions</b>	250 x 3 mm
<b>Order number</b>	25CE200ESJ

## Recommended instrumentation

This application requires an isocratic HPLC system equipped with degasser, autosampler, column oven, and UV detector. Other configurations are also available. Please contact KNAUER to configure a system that's perfect for your needs.

Description	Order No.
Smartline Pump 1050, incl. 10 ml pump head	A50353-1
Smartline Manager 5050 with LPG and degasser	A5333
SmartMix static mixer	A5351
Autosampler 3950	A5005V1
Smartline Oven 4050	A5300
Smartline PDA Detector 2850	A5271
10 mm flow cell	A4130
ChromGate software	A1493

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