DETERMINATION OF CINEOLE IN ESSENTIAL OILS

Principle of method

Cineole determination in essential oils, especially in eucalyptus oil is performed on gas chromatograph by use of furfurol as internal standard.

Reagents

Cineole.: Standard pure compound

Furfurol.: Standard pure compound.

Instrumentation

Gas chromatograph: Equipped with Flame Ionization Detector, glass column (4 mm ID, 2 m length) with Carbowax 20 M 10% on Chromosorb W-AW-DMCS 80-100 mesh.

Working Conditions on GC.: Gas flow rates: N_2: 30 mL/min; H_2: 30 mL/min and Air: 300 mL/min.

Temperatures.: Detectors: 225^{o}C; Injection Ports: 160^{o}C; Column Oven: 110-180^{o}C.

Experimental

Injection Precision

0.1 µL of cineole is injected to the GC for 20 times. Relative standard deviation between responses should be lower than 0.5%.

CINEOLE				FURFUROL			
Injection No	Response	Injection No	Response	Injection No	Response	Injection No	Response
1		11		1		11	
2		12		2		12	
10		20		10		20	
N= 20	SD=	RSD= %		N=20	SD=	RSD= %	

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Detector Responses

Known amounts of pure cineole and furfurol are injected to the GC at least 10 times and the corresponding responses are obtained. The curves are plotted for the responses versus injected volumes.The linear part of the curve is used to discuss the limit of determination.

CINEOLE		FURFUROL	
Volume injected (µL)	Response	Volume injected (µL)	Response
0.02		0.02	
0.03		0.03	
0.05		0.05	
0.08		0.08	
0.10		0.10	
0.15		0.15	
0.20		0.20	
0.25		0.25	
0.30		0.30	

Preliminary GC Work

Retention time of cineole is determined from earlier experiments.

Essential oil (1 µL) is injected to GC. Possible cineole presence is discussed comparing the retention times of the peaks.

If the cineole presence is doubtful, known amount of cineole is added to the known amount of sample and this mixture is injected.

Furfurol is injected to the GC in order to check its position in the chromatogram. Furfurol peak should be clear and isolated.

Use densities of both cineole and furfurol in calculations.

Calibration Curves

Standard calibration curves obtained from pure cineole and cineole +internal standard mixtures with different weight ratios are prepared by the application of known mixtures to GC.

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Related response ratios are calculated from the chromatograms and response ratio (R_f= response of cineole/response of internal Standard) versus weight ratio (W_r= weight of cineole/weight of furfurol) are plotted. A linear relation should be observed passing through origin.

Data for Calibration Curve

Cineole % (in weight) in the mixture	W_r = weight of cineole/weight of furfurol	R_r = response of cineole/ response of furfurol
20		
30		
40		
50		
60		
70		
75		
80		

Cineole analysis in essential oil

Known amounts of furfurol, e.g., 0.2000 g, 0.5000 g and 0.7500 g, are mixed well with known amount of essential oil (e.g., 1.0000 g). The mixtures are injected to the GC. From the calibration curve, response ratio of cineole and furfurol gives the weight ratio of cineole and furfurol. The cineole weight is calculated from the weight ratio. The ratio of the cineole weight to the essential oil weight gives the percent abundance of the cineole. Arithmetic mean of three independent injections is reported.

W_r = Weight of cineole/ weight of furfurol C_1= (Weight of cineole/Weight of essential oil)x100 =…..% cineole w/w.

$C_{mean} = (C_1+C_2+C_3)/3$ = ……. g/g (if necessary, confidence interval is reported with 95% confidence level)

REFERENCE

Aydın, A., Mutlu, A., and Arınel, N.:Gas chromatographic studies on the determination of cineole content in the eucalyptus oil, ***Chimica Acta Turcica***, 5 (1977) 191-200.

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