

Gas Chromatography Analytical Methods for Teaching Laboratories

Separation of the components of commercially available spirits

using different column phases and GC FID



Introduction

Organic solvents, such as methylated spirits, white spirits, and turpentine, are routinely used around the home for cleaning, thinning paint, cleaning paint brushes or as fuel. These are not pure solvents and may contain impurities or additives. Methylated spirits, for example, is mainly ethanol, but to prevent recreational consumption and the addition of taxes applied to alcoholic beverages, compounds such as methanol and pyridine are added to make it poisonous, bad tasting and foul smelling.

This application utilizes a GC with a standard inlet and flame ionization detector to analyze the components found in one or more commercially available spirits. The samples will be analyzed multiple times using different oven temperature programs and column phases to demonstrate how temperature and column phase can affect analyte retention.

Reagents

N-methyl-2-pyrrolidone – analytical grade

Sample material – any commercially available spirit, e.g. methylated spirits, or paint thinners.

Individual solvent standards may be prepared from appropriate laboratory stocks to aid identification of the different components found in the test samples.

Safety

Lab coats, safety glasses and gloves should be worn when handling reagents.

Follow all appropriate safe operating procedures and beware of any specific actions raised in the associated risk assessments.

Apparatus

- Gas chromatograph fitted with a split/splitless injection port and flame ionization detector (FID).
- Autoinjector and autosampler capable of handling liquid samples in 1.5 mL vials
- SH-1 column, 60 m x 0.32 mm I.D. x 1.0 μ m, p/n 227-36099-02 (or suitable equivalent).
- SH-624 column, 60 m x 0.32 mm I.D. x 1.8 μ m, p/n 221-75864-60 (or suitable equivalent).
- SH-Wax column, 60 m x 0.32 mm I.D. x 1.0 μ m, p/n 227-75897-60 (or suitable equivalent).
- 1.5 mL autosampler vials and caps.
- Appropriate laboratory glassware for the preparation of the test samples.

Instrument Conditions

GC Parameters	
Injection Temperature	250°C
Injection Mode	Split
Split Ratio	1:10
Purge flow	3 mL/min
Column	See above
Oven Program (suggestion for initial method)	40°C, hold for 0 minutes, 4°C/min to 250°C, hold for 0 minutes
Carrier Gas	Helium, linear velocity 25 cm/s
Injection Volume	1.0 µL
FID Temperature	280°C
Hydrogen Flow rate	32 mL/min
Air flow rate	200 mL/min
Make up gas (N ₂) flow rate	24 mL/min
Sampling Rate	40 msec

Preparation of individual solvent standards

Dilute individual laboratory solvents with N-methyl-2-pyrrolidone in a volumetric flask to give a final concentration of approximately 10 mg/L. Then transfer 1 mL in to a 1.5 mL vial for analysis.

Preparation of test sample

Dilute the test sample in N-methyl-2-pyrrolidone to give a final concentration of approximately 50mg/L. Then transfer 1mL transfer in to a 1.5 mL vial for analysis.

Interpretation of results

1. SH-1 column

The components of the test sample should be adequately separated to allow each individual peak to be integrated. The oven ramp rate can be experimented with to see how it will affect analyte retention. In most cases, slowing it down will help to move compounds apart and speeding it up will move them closer together.

The example below shows a mixture of 85 organic solvents analyzed on an SH-1 column. Most of the components are resolved, however, there are some coeluting peaks that have not been separated sufficiently, e.g. acetaldehyde and methanol.

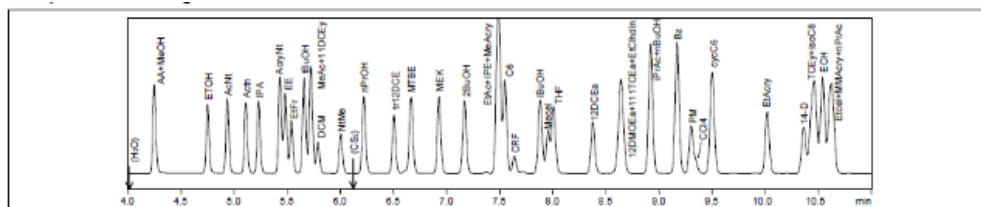


Fig.2 4.0 to 11.0 min

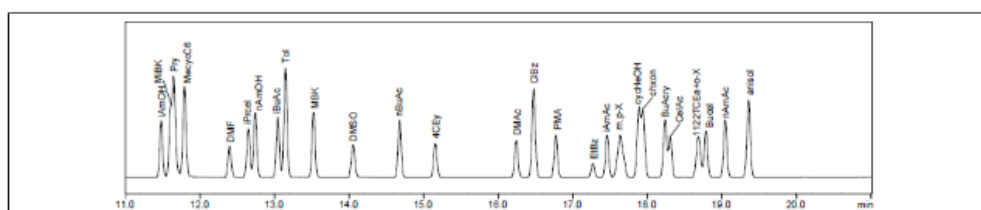


Fig.3 11.0 to 21.0 min

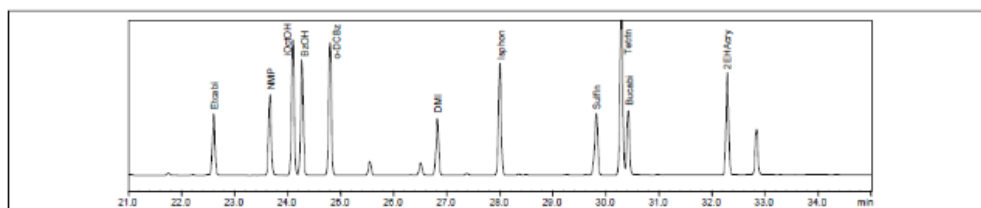


Fig.4 21.0 to 35.0 min

2. SH-624 column

The example below shows the same mixture of 85 organic solvents analyzed on an SH-624 column. The retention times of the components have changed and now acetaldehyde and methanol have separated.

3. SH-WAX column

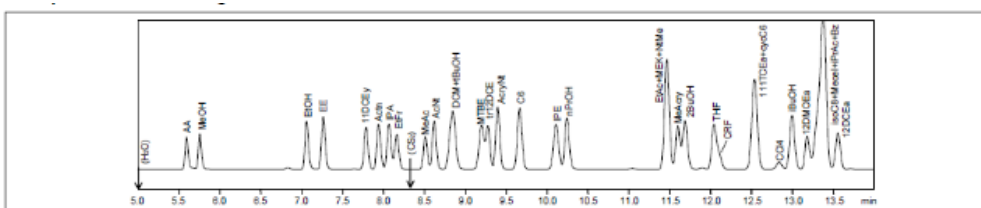


Fig.2 5.0 to 14.0 min

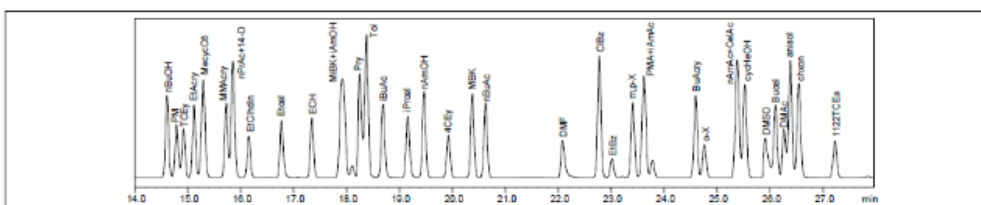


Fig.3 14.0 to 28.0 min

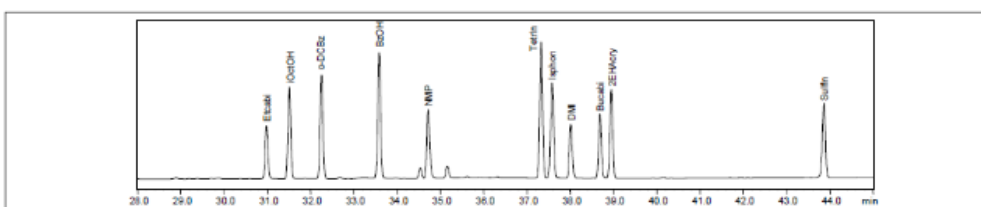
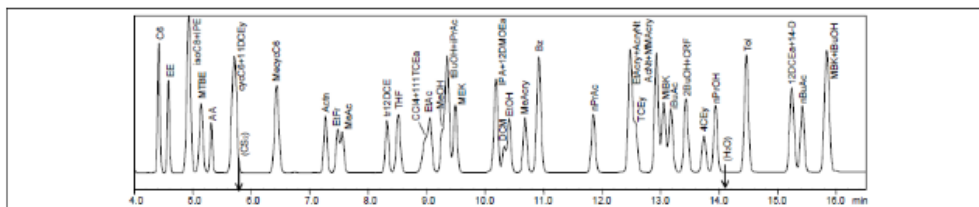


Fig.4 28.0 to 45.0 min

The mixture of 85 organic solvents was analyzed on a third column phase. Again, the retention times have changed for the sample components with some separations improving and others getting worse.



Notes

The instrument conditions are based on the Shimadzu application notes G241 'Analysis of Organic Solvents using Capillary Column Rtx-1', G242 'Analysis of Organic Solvents using Capillary Column Rtx-624' and G243 'Analysis of Organic Solvents using Capillary Column Rtx-WAX'. If an alternative column or carrier gas is used, these conditions will need to be adapted appropriately.

Table 2 List of Organic Solvents (with Abbreviations)

Organic solvent name	Abbreviation	Organic solvent name	Abbreviation	Organic solvent name	Abbreviation
Acetaldehyde	AA	Ethyl Formate	EtFr	Hexane	C6
Methanol	MeOH	Methyl Acetate	MeAc	Isooctane	isoC8
tert-Butanol	tBuOH	Ethyl Acetate	EtAc	Cyclohexane	cycC6
Isopropanol	IPA	Isopropyl Acetate	iPrAc	methylcyclohexane	MecycC6
Ethanol	EtOH	n-Propyl Acetate	nPrAc	Benzene	Bz
sec-Butanol	2BuOH	Isobutyl Acetate	iBuAc	Toluene	Tol
n-Propanol	nPrOH	n-Butyl Acetate	nBuAc	Ethylbenzene	EtBz
Isobutanol	iBuOH	Isoamyl Acetate	iAmAc	p-Xylene	p-X
n-Butanol	nBuOH	n-Amyl Acetate	nAmAc	m-Xylene	m-X
Isoamyl Alcohol	iAmOH			o-Xylene	o-X
n-Amyl Alcohol	nAmOH	Methyl Acrylate	MeAcry	Tetralin	TetrIn
Cyclohexanol	cycHeOH	Ethyl Acrylate	EtAcry		
Isooctanol	iOctOH	Acrylonitrile	AcryNt	Pyridine	Pry
Benzyl Alcohol	BzOH	Acetonitrile	AcNt	N,N-Dimethylformamide	DMF
		Methyl Methacrylate	MMAcry	N,N-Dimethylacetamide	DMAc
Ethyl Ether	EE	Nitromethane	NtMe	Dimethyl Sulfoxide	DMSO
Isopropyl Ether	IPE	Butyl Acrylate	BuAcry	N-Methylpyrrolidone	NMP
tert-Butyl Methyl Ether	MTBE	2-Ethylhexyl Acrylate	2EHAcry	1,3-Dimethyl-2-Imidazolidinone	DMI
Tetrahydrofuran	THF				
1,4-Dioxane	14-D	1,1-Dichloroethylene	11DCEy	1,2-Dimethoxyethane	12DMOEa
Anisole	anisol	trans-1,2-Dichloroethylene	tr12DCE	Propylene Glycol Monomethyl Ether	PM
Acetone	Actn	Tetrachloromethane	CCl4	Ethylene Glycol Monomethyl Ether	Mecel
Methyl Ethyl Ketone	MEK	1,1,1-Trichloroethane	111TCEa	Propylene Glycol Monomethyl Ether Acetate	PMA
Methyl Isobutyl Ketone	MIBK	Dichloromethane	DCM	Ethylene Glycol Monoethyl Ether	Etcel
Methyl Butyl Ketone	MBK	Trichloroethylene	TCEy	Ethylene Glycol Monoisopropyl Ether	iPcel
Cyclohexanone	chxon	Chloroform	CRF	Ethylene Glycol Monoethyl Ether Acetate	CelAc
Isophorone	Isphon	tetrachloroethylene	4CEy	Ethylene Glycol Monobutyl Ether	Bucel
Sulfolane	Sullfn	1,2-Dichloroethane	12DCEa	Diethylene Glycol Monoethyl Ether	EtCabi
		Epichlorohydrin	ECH	Diethylene Glycol Monobutyl Ether	Bucabi
		Chlorobenzene	ClBz		
		Ethylenechlorohydrin	EtClndIn		
		1,1,2,2-Tetrachloroethane	1122TCEa	Water	H ₂ O
		o-Dichlorobenzene	o-DCBz	Carbon Disulfide	CS ₂
				acetic acid	