

COMPREHENSIVE ANALYSIS OF THREE METHODS IN DETERMINATION OF VOLATILE COMPOUNDS IN SPIRIT DRINKS

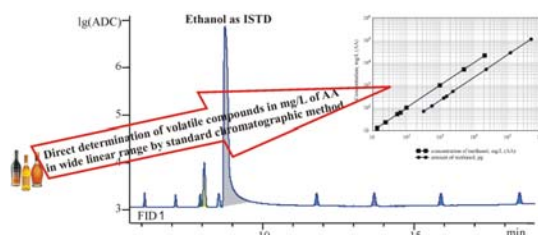
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➤ Proposed new method of using ethanol as internal standard ("ethanol as IS") in gas chromatographic analysis of volatile compounds in spirit drinks [1] is investigated experimentally from different sides in comparison with traditional internal standard (IS) method and external standard (ES) method.

➤ Further verification and validation of the method "ethanol as ISTD" was accomplished on different gas chromatographs in three testing laboratories of Belarus and Russia.

➤ This method provides determination of volatile compound concentrations in spirit drinks expressed directly in milligrams per liter of absolute alcohol (mg/L (AA)) without quantitative measuring the alcohol content of the analyzed sample.



[1] Charapitsa S.V. et al. Direct Determination of Volatile Compounds in Spirit Drinks by Gas Chromatography. *Journal of Agricultural and Food Chemistry*, 2013, **61**, 2950-2956.

Methods and Materials

All individual standard compounds were purchased from Sigma-Fluka-Aldrich (Berlin, Germany). The standard solutions were prepared by adding the individual standard compounds to the ethanol-water mixture (96:4) by weight. High-grade ethanol was purchased from Minsk-Kristall Winery and Distillery Plant (Minsk, Belarus). The ethanol-water (96:4) solutions GSO-8404 and ethanol-water (40:60) GSO-8405 solutions were purchased from VNIIPBT (Moscow, Russia). Seven standard ethanol-water (96:4) solutions of volatile compounds (VC) were prepared gravimetrically. The initial standard solution, VC-1, was prepared by adding the individual compounds to high-grade ethanol in accordance with recommendations of ASTM D 4307. Subsequent standard solutions, VC-2 – VC-7, were prepared by adding VC-1 to high-grade ethanol.

Table. Concentrations of analyzed volatile compounds are expressed in mg/L (AA), 1-pentanol was introduced as internal standard

Compound	Concentration, mg/L (AA)							Relative error % (P=0,95)
	VC-1	VC-2	VC-3	VC-4	VC-5	VC-6	VC-7	
acetaldehyde	4275	1096	111	56,2	11,2	2,22	1,13	± 3 %
methyl acetate	4397	1128	114	57,8	11,5	2,29	1,17	± 3 %
ethyl acetate	4173	1070	108	54,9	10,9	2,17	1,11	± 3 %
methanol	41995	10774	1092	555,5	113,3	24,96	14,3	± 3 %
2-propanol	3991	1025	105	54,1	12,1	3,69	2,70	± 3 %
1-propanol	4012	1029	104	52,8	10,5	2,08	1,06	± 3 %
isobutyl alcohol	3975	1020	103	52,3	10,4	2,06	1,05	± 3 %
n-butanol	4071	1044	106	53,5	10,7	2,11	1,08	± 3 %
isoamyl alcohol	4071	1044	106	53,5	10,7	2,11	1,08	± 3 %
1-pentanol (IS)	27,1	27,1	27,1	27,1	27,1	27,13	27,13	± 3 %

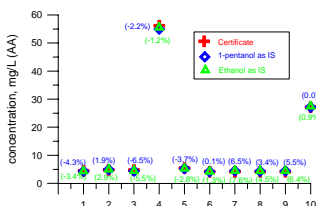
Analytical characteristics of the response factors

Compound	1-pentanol as IS		ES		Ethanol as IS		LOD* (mg/L)
	RRF	Correlation coefficient, R ²	RF (mg/L)/(pA*min)	Correlation coefficient, R ²	RRF	Correlation coefficient R ²	
acetaldehyde	2,396	0,9997	266,1	0,9997	1,710	0,9997	0,344
methyl acetate	2,491	0,9997	276,7	0,9996	1,779	0,9999	0,683
ethyl acetate	1,757	0,9997	195,1	0,9997	1,254	0,9999	0,322
methanol	2,133	0,9998	236,9	0,9997	1,523	0,9999	0,231
2-propanol	1,400	0,9998	155,5	0,9997	0,999	0,9999	0,119
ethanol	1,413	N/A	155,5	N/A	1	N/A	N/A
1-propanol	1,179	0,9997	130,9	0,9996	0,841	0,9999	0,222
isobutyl alcohol	1,018	0,9998	113,0	0,9997	0,727	0,9999	0,178
n-butanol	1,117	0,9999	124,1	0,9998	0,798	0,9999	0,189
isoamyl alcohol	1,030	0,9999	114,4	0,9998	0,735	0,9999	0,179
1-pentanol	1	N/A	110,1	N/A	0,708	N/A	0,271

* limit of detection (LOD)

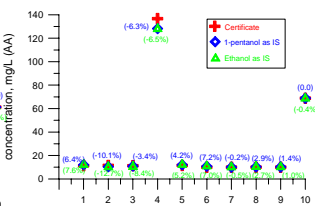
Experimental measured concentrations of volatile compounds in solutions VC-4, VC-5 and VC-6 with (Relative discrepancy, %) for three methods: 1-pentanol as IS, ES and Ethanol as IS in comparison with data by gravimetric method

Experimental measured concentrations of volatile compounds in the ethanol-water (40:60) solutions GSO 8405 with (Relative discrepancy, %)

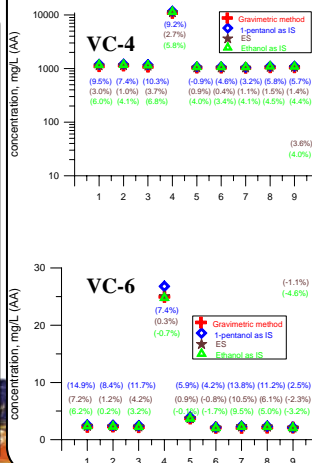


- 1 - acetaldehyde
- 2 - methyl acetate
- 3 - ethyl acetate
- 4 - methanol
- 5 - 2-propanol

Experimental measured concentrations of volatile compounds in the ethanol-water (96:4) solutions GSO 8404 with (Relative discrepancy, %)



- 6 - 1-propanol
- 7 - isobutyl alcohol
- 8 - n-butanol
- 9 - isoamyl alcohol
- 10 - 1-pentanol (IS)



The results obtained in testing laboratories of Belarus and Russia using the three methods well agreed.

- 1 - acetaldehyde
- 2 - methyl acetate
- 3 - ethyl acetate
- 4 - methanol
- 5 - 2-propanol
- 6 - 1-propanol
- 7 - isobutyl alcohol
- 8 - n-butanol
- 9 - isoamyl alcohol
- 10 - 1-pentanol (IS)