

NCIRFAT

食品真实性技术国际联合研究中心

NATIONAL CENTER OF INTERNATIONAL RESEARCH
ON FOOD AUTHENTICITY TECHNOLOGY



葡萄酒真实性鉴别技术研究与应用

Research and Application of Wine Authenticity
Identification Technology

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➤ Background 背景

- 国际葡萄酒及烈酒研究所(IWSR)预计中国将在未来成为全球第二大葡萄酒市场，2021年前销量增至230亿美元。

International Wine and Spirits Research Institute (IWSR) predict China will become the world's second-largest wine market and the sales in China will have increased to \$23 billion by 2021.

- 假冒伪劣葡萄酒产品的威胁 Threat of counterfeit wines
- 葡萄酒行业亟待政府监管 Wine industry needs supervision



Fake wines always be found and reported.

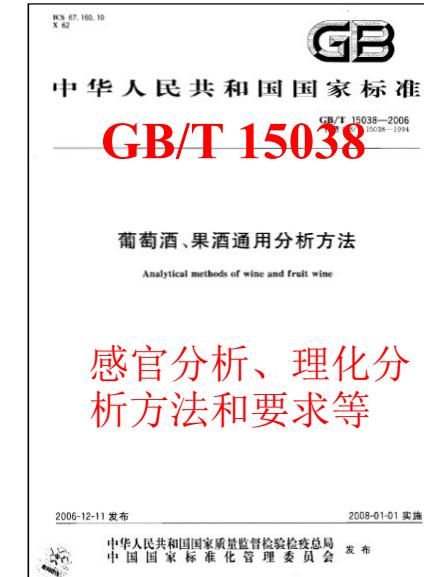


➤ China Wine Standard 中国葡萄酒标准

- GB 15037-2016 Wine

**Strict definition and classification following OIV standard
as a reference, high requirements in safety and quality**

- GB/T 15038-2006 Wine, Fruit wine analytical method (sensory, phy-chemical character)
- GB/T 11856-2008 Blanky
- GB/T 23543-2009 Good hygiene practice for wine industry
- GB/T 25504-2010 Ice wine





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➤ China Wine Standard 中国葡萄酒标准

- Guideline for the identification of varietal wines
- Guideline for the detection of water addition in wine
- Guideline for the identification of original wine

ICS 67.160.10
X 62



中华人民共和国国家标准

GB/T XXXXX—XXXX

葡萄酒掺水识别技术导则

Guidance for the identification of adulterate wine with water

点击此处添加与国际标准一致性程度的标识

(报批稿)

(本稿完成日期：20160515)

XXXX - XX - XX 发布

XXXX - XX - XX 实施

中华人民共和国国家质量监督检验检疫总局
中国国家标准化管理委员会发布

ICS 67.160.10
X 62



中华人民共和国国家标准

GB/T XXXXX—XXXX

产地葡萄酒识别技术导则

Guideline for Distinguish original wine

点击此处添加与国际标准一致性程度的标识

XXXX - XX - XX 发布

XXXX - XX - XX 实施

中华人民共和国国家质量监督检验检疫总局
中国国家标准化管理委员会发布

202X - XX - XX 发布

202X - XX - XX 实施

国家市场监督管理总局发布
国家标准化管理委员会发布



中华人民共和国国家标准

GB/T XXX—202X

品种葡萄酒识别技术导则

Guideline for the identification of varietal wines

(报批稿)

XXXX - XX - XX 发布

XXXX - XX - XX 实施

中华人民共和国国家质量监督检验检疫总局
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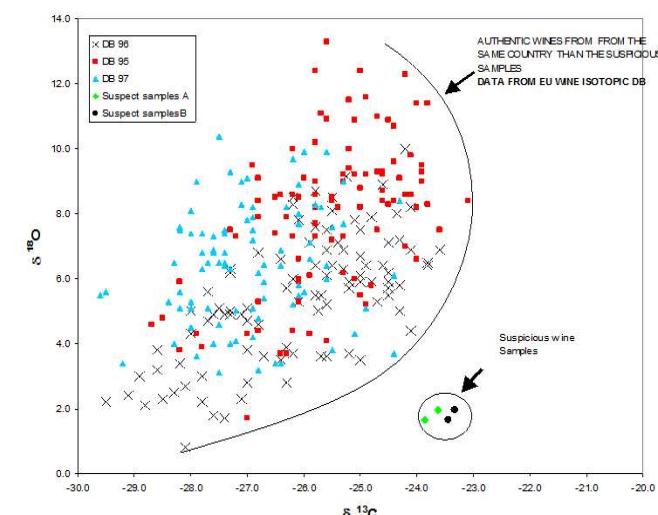
➤ EU Wine Standard 欧盟葡萄酒标准

Year	Method	compound		同位素
1987	OIV, recueil des méthodes d'analyse	ethanol	SNIF-NMR	(D/H)I (D/H)II
1990	EC regulation 2676/90, annex 8	ethanol	SNIF-NMR	(D/H)I (D/H)II
1996	OIV Resolution OENO 2/96	water	IRMS	18O/16O
1997	EC Regulation No. 822/97	water	IRMS	18O/16O
2001	OIV Resolution OENO 17/2001	ethanol	IRMS	13C/12C
2003	EC No 440/2003, annex 2	ethanol	IRMS	13C/12C
2005	OIV Resolution OENO 7/2005	CO2	IRMS	13C/12C
2009	OIV Resolution OENO 353/2009	water	IRMS	18O/16O
2009	OIV Resolution OENO 381/2009	ethanol	IRMS	13C/12C
2010	OIV Resolution OENO 343/2010	glycerol	IRMS	13C/12C

EC Reg N° 555/2008



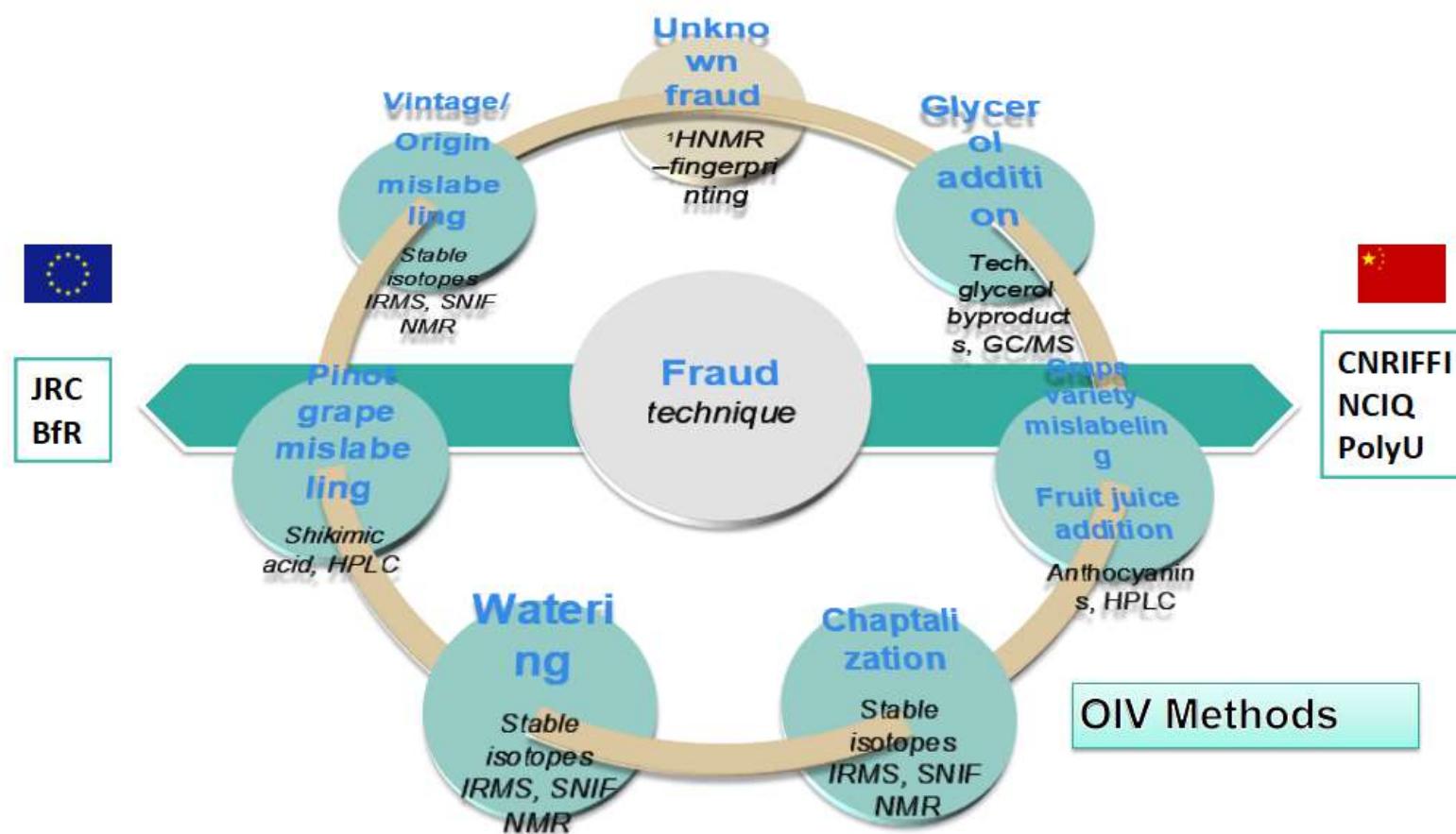
Use of the EU Wine Databank



➤ 中欧食品安全合作项目 EU-China-Safe project



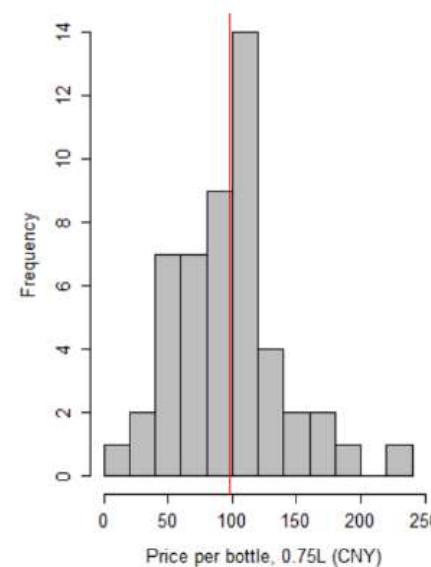
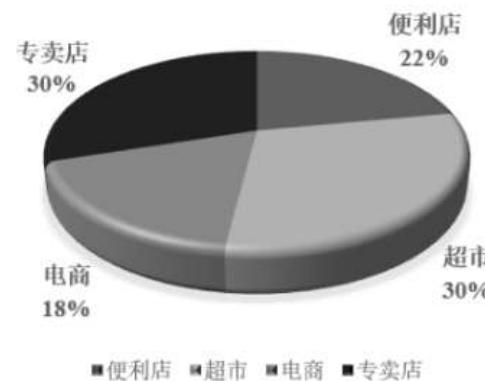
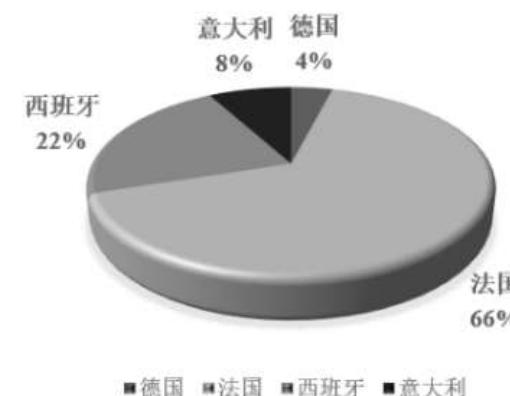
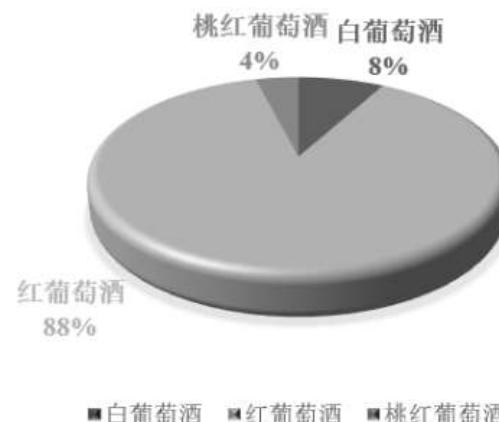
➤ 葡萄酒造假形式 Wine Counterfeiting



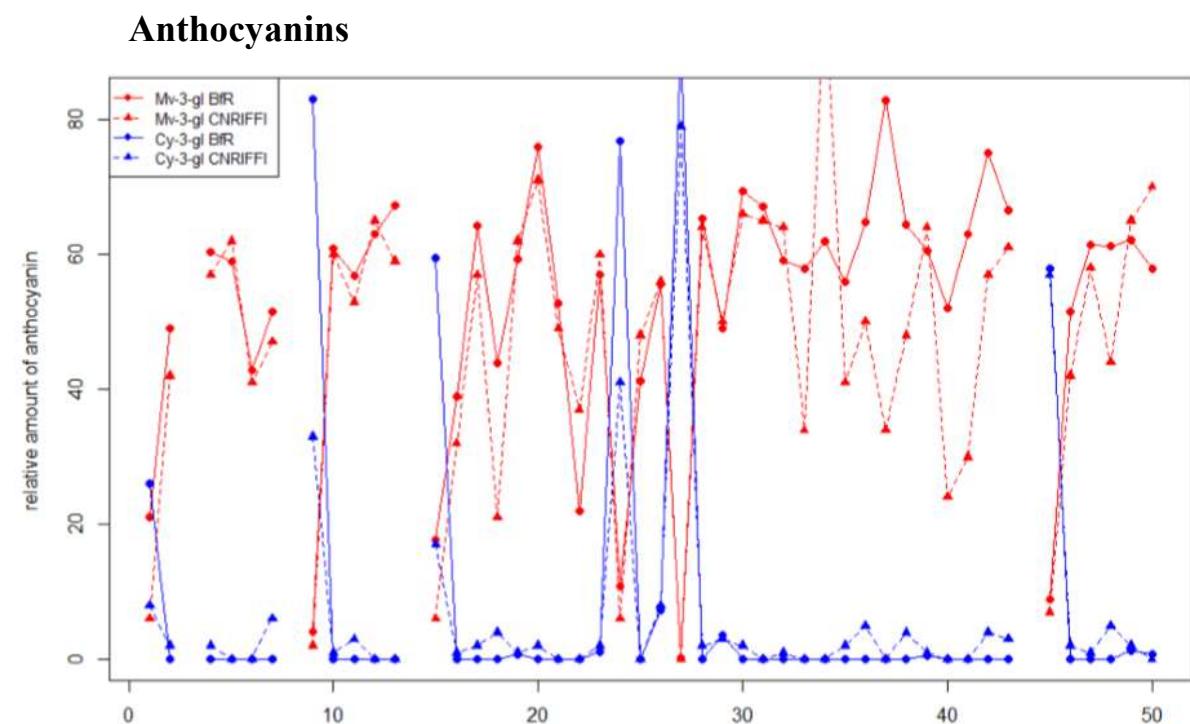
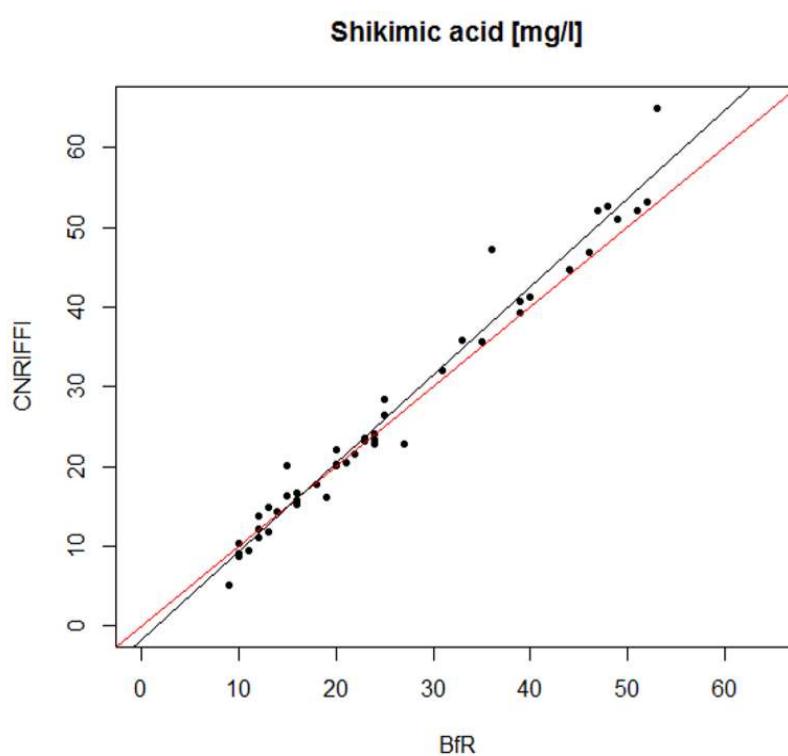
Wine Methods Investigation, Transfer and Exchange

Number	BfR JRC	CNRIFI NCIQ Polyu	Target
1	Determination of 18O/16O isotope ratio of wine water by IRMS (OIV-MA-AS2-12)	QBT 4853-2015	Watering Chaptalisation Origin
2	Determination of 13C/12C isotope ratio of wine ethanol by IRMS (OIV-MA-AS312-06)	QBT 5164-2017	
3	Determination of by-products of technical glycerol in wine by GC-MS (OIV-MA-AS315-15)	OIV-MA-AS315-15	Technical glycerol addition
4	Determination of shikimic acid in wine by HPLC-UV (OIV-MA-AS313-17)	OIV-MA-AS313-17	Pinot grape mislabeling
5	Determination of major anthocyanins in wine by HPLC (OIV-MA-AS315-11)	OIV-MA-AS315-11	Grape variety Fruit juice addition
6	Determination of deuterium distribution in wine ethanol by SNIF-NMR (Method OIV-MA-AS311-05)	OIV-MA-AS311-05	Watering Chaptalisation Origin
7	Non-targeted wine analysis by ¹ H NMR (Bruker WineScreener method (Godelmann et al.,2013))	Bruker WineScreener	Unknown Fraud

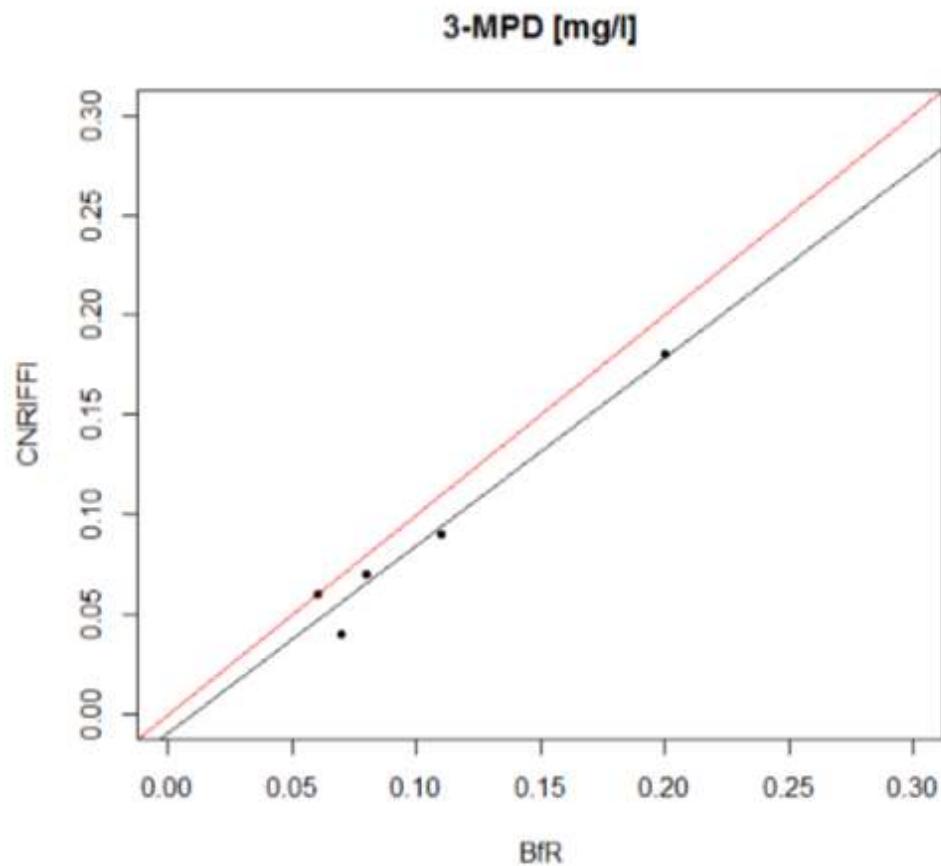
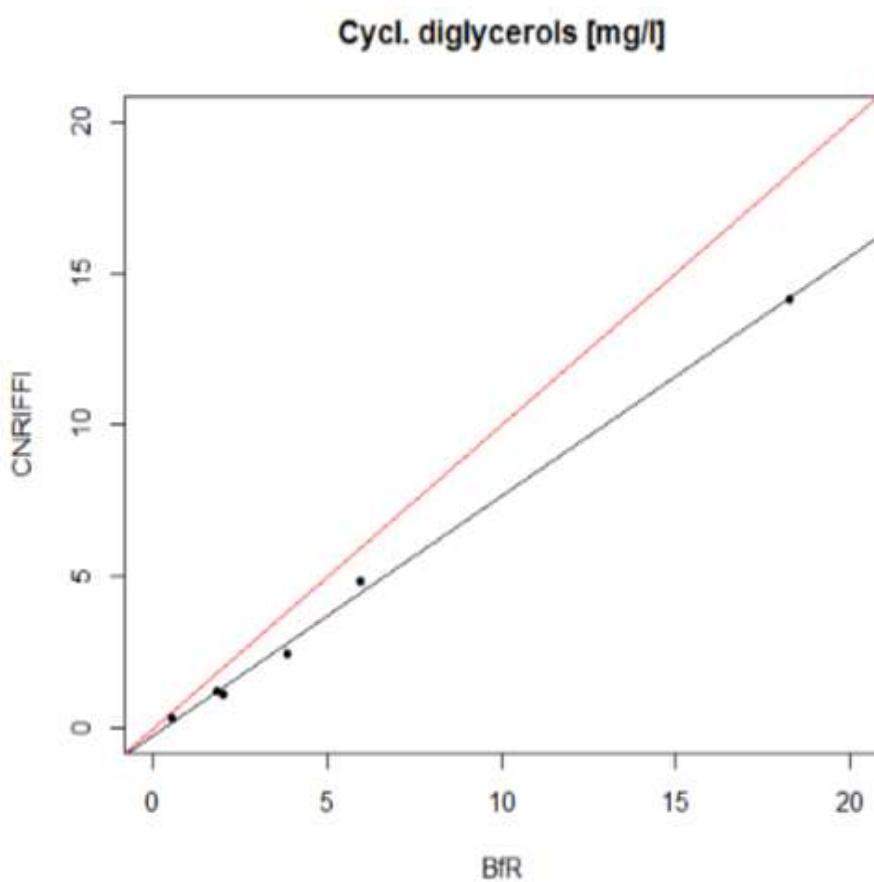
➤ Wine sampling 葡萄酒样品



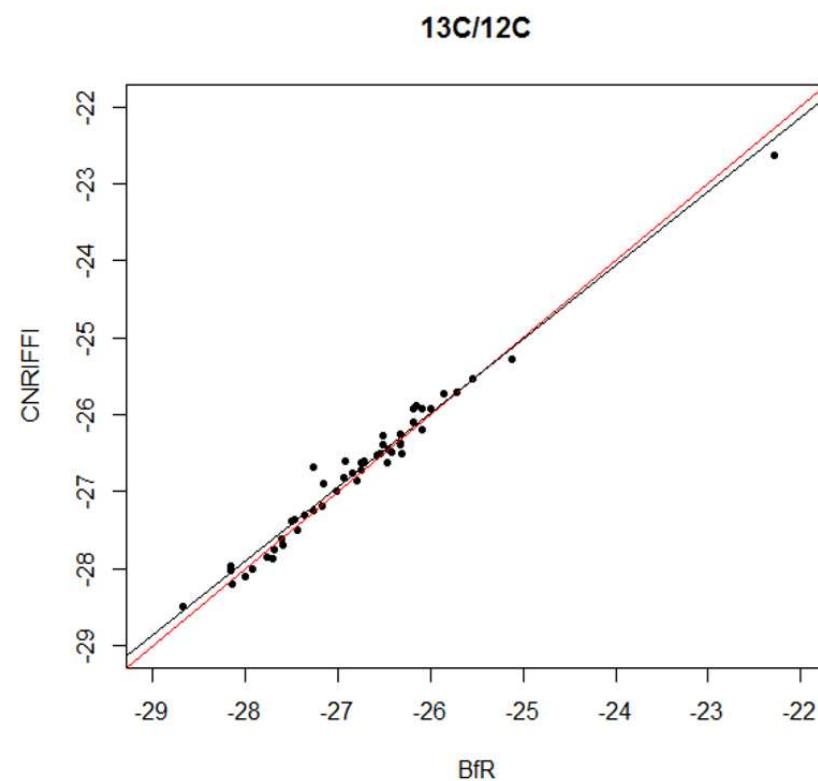
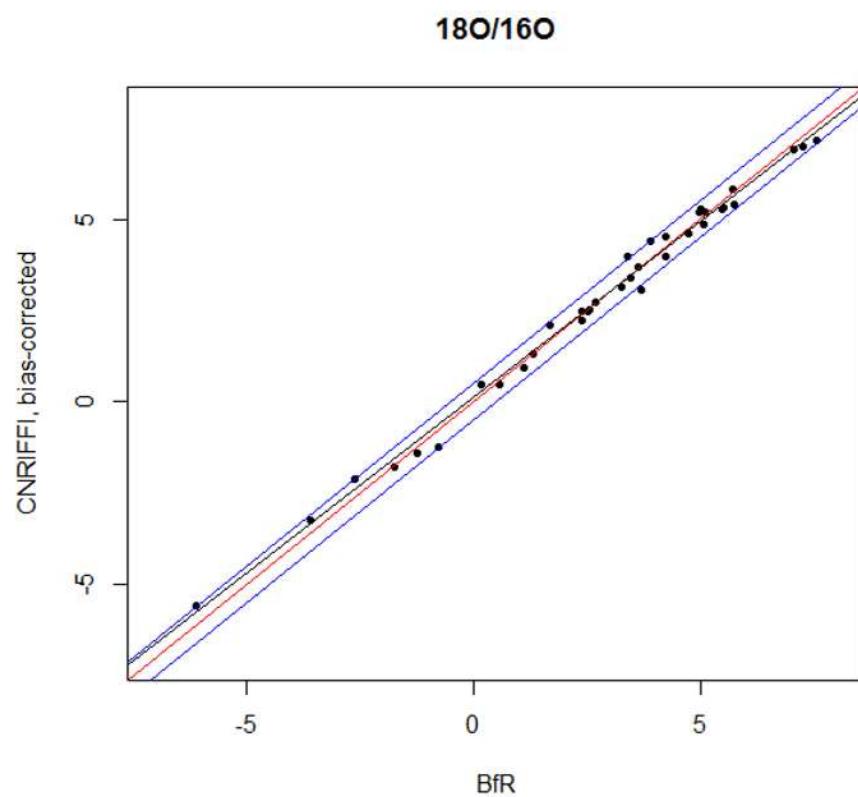
➤ Wine Methods Verification Results 葡萄酒分析方法验证结果



➤ Wine Methods Verification Results 葡萄酒分析方法验证结果

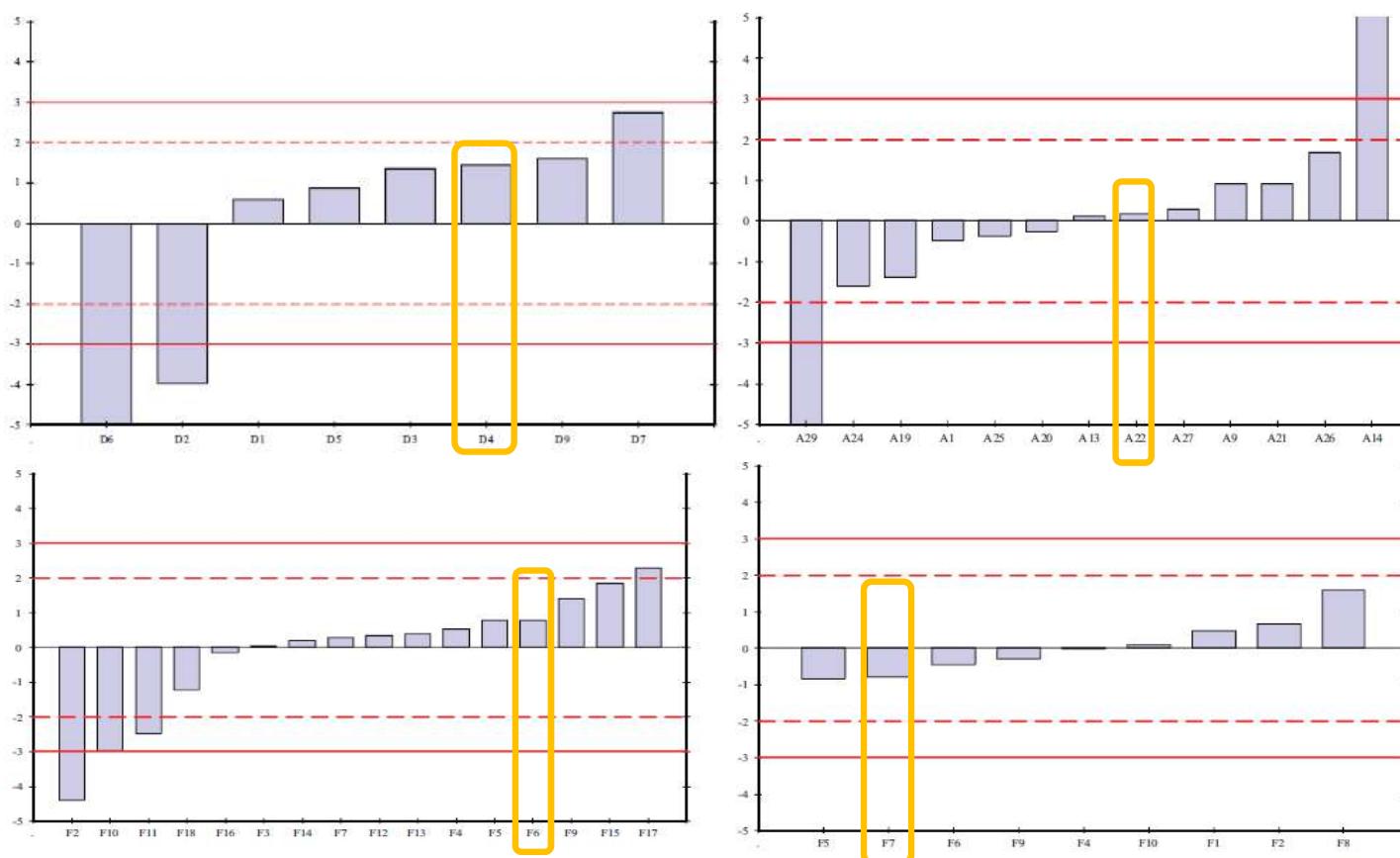


➤ Wine Methods Verification Results 葡萄酒分析方法验证结果



➤ Wine Methods Verification Results 葡萄酒分析方法验证结果

Determination of $^{18}\text{O}/^{16}\text{O}$ isotope ratio of wine water by IRMS for **FIT-PTS**
(Chinese Standard)



ICS 67.080.10
分类号: X50
备案号: 52205-2015

QB

中华人民共和国轻工行业标准

QB/T 4853—2015

葡萄酒中水的稳定氧同位素比值 ($^{18}\text{O}/^{16}\text{O}$)
测定方法 同位素平衡交换法

Determination of the oxygen isotope ratio ($^{18}\text{O}/^{16}\text{O}$) of water in wines
—Method using isotopic equilibrium exchange reaction

2015-10-10 发布

2016-03-01 实施

中华人民共和国工业和信息化部 发布

➤ Wine Methods Innovation 葡萄酒分析方法创新

Determination of $^{13}\text{C}/^{12}\text{C}$ isotope ratio of wine ethanol by IRMS

	Chinese standard method (QBT 5164-2017)	OIV method (OIV-MA-AS312-06)
Equipment	GC-C-IRMS	Ethanol distillation unit, EA-IRMS
Pretreatment	Wine sample diluted with acetone, no distillation, injected into CP wax column	Pure ethanol extracted from wine sample,
Sample volume	70 uL wine (10% vol)	300 mL wine(10% vol)
Regent	1 mL Acetone (HPLC grade)	none
Analysis time	1 minute for dilution and 20 minutes	4 hours for distillation and 12 minutes

➤ Wine Methods Verification Results 葡萄酒分析方法验证结果

Determination of deuterium distribution in wine ethanol by SNIF-NMR (OIV-MA-AS311-05)

ETHANOL

SAMPLE NUMBER:18070001

Spectrometer Filename: Nb Spectra : 10

Preparation Parameters:

Product Weight : 2.61630g Purity: 0.8904
Reference Weight: 1.30120g Reference name:317P(153.70ppm)

Treatment:

Type of processing :EthanolReprocessing

Results:

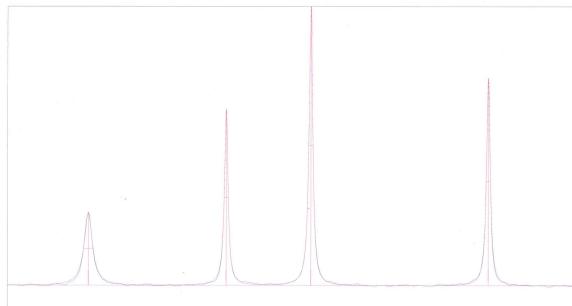
$(D/H)_i$ Intensity in ppm

	$(D/H)_i$	$(D/H)_2$
1	101.50	128.6
2	101.90	127.88
3	101.71	128.30
4	101.21	127.80
5	101.92	127.76
6	101.97	128.35
7	101.66	128.4
8	102.29	128.63
9	101.32	129.16
10	101.27	127.97
Mean	101.59	128.22
SD	0.45	0.45

Ratio R (Intensity)

Mean : 2.524 (0.013)

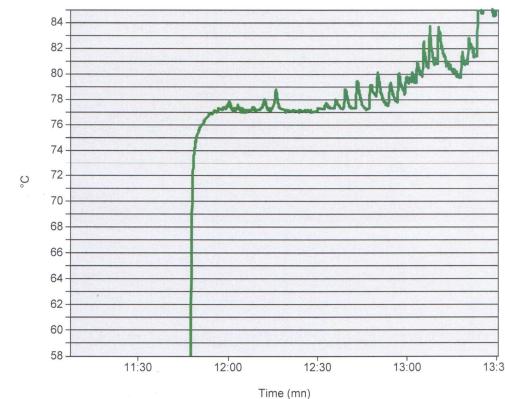
Peakwidth
CH₃ TMU
1 2.423 2.426



A.D.C.S. 18070001 7/16/2018 10:58:55 AM

Description: Column # 1 Op: NCIQ

Tare (g):	160.20	Wine temperature (°C):	20
V (ml):	200.00	Wine weight (g):	197.6
Tare + V (g):	357.76	Residue weight (g):	175.5
S = Tare + residue (g):	335.74	Distillate weight (g):	21.5
Empty Erlen (g):	57.02	Weight loss (g):	0.5
Erlen + distillate (g):	78.52		
Wine density (g/cm3):	0.9878	pH:	0
Wine alcoholic % volume, tQ (%):	12.5		
Distillate alcoholic grade % w/w (KF), tD (%):	89.0		
Alcohol quantity (>10g) (g):	19.14	Curve type (A,B,C):	
Weight loss (g):	0.52	Distillation yield (>90%) (%):	97.0
tD (>85%) (%):	89.04		
Validity level (<2):	0		

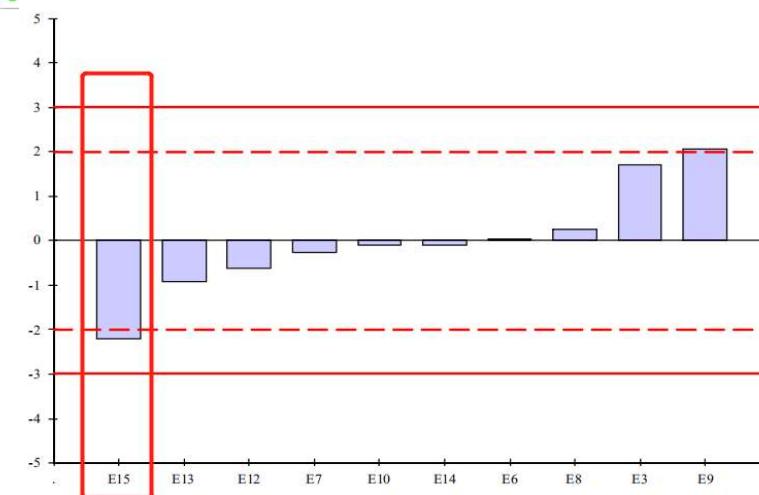
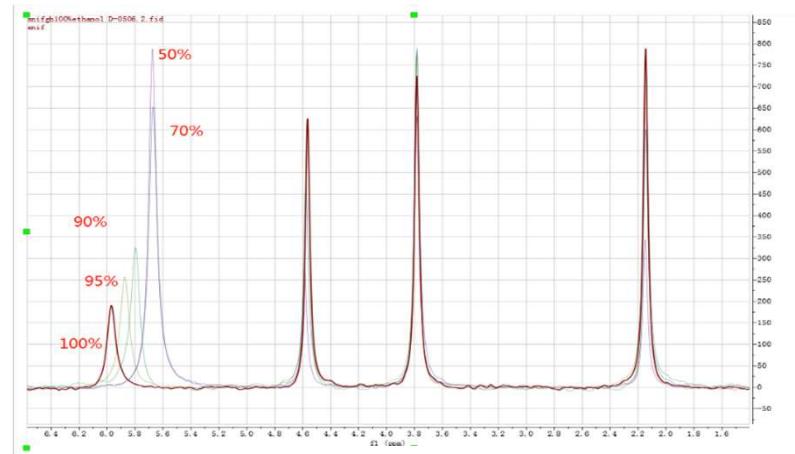
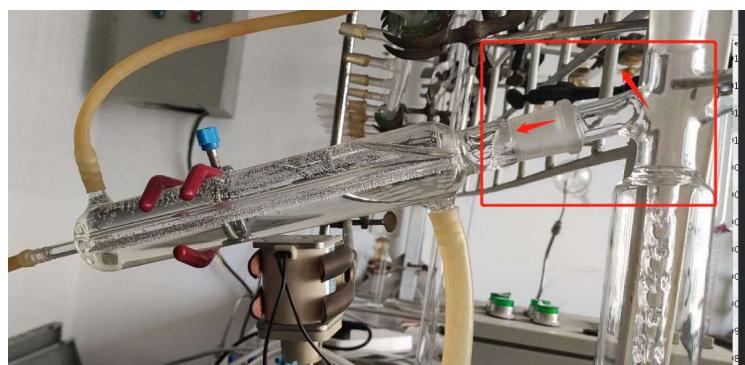
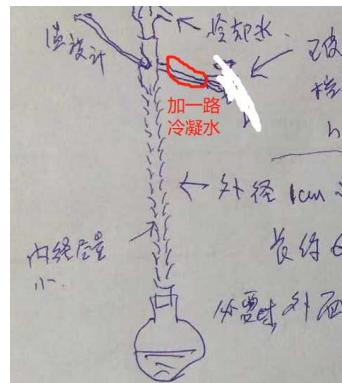


BfR and CNRIFFI Comparsion Results 样品比对结果

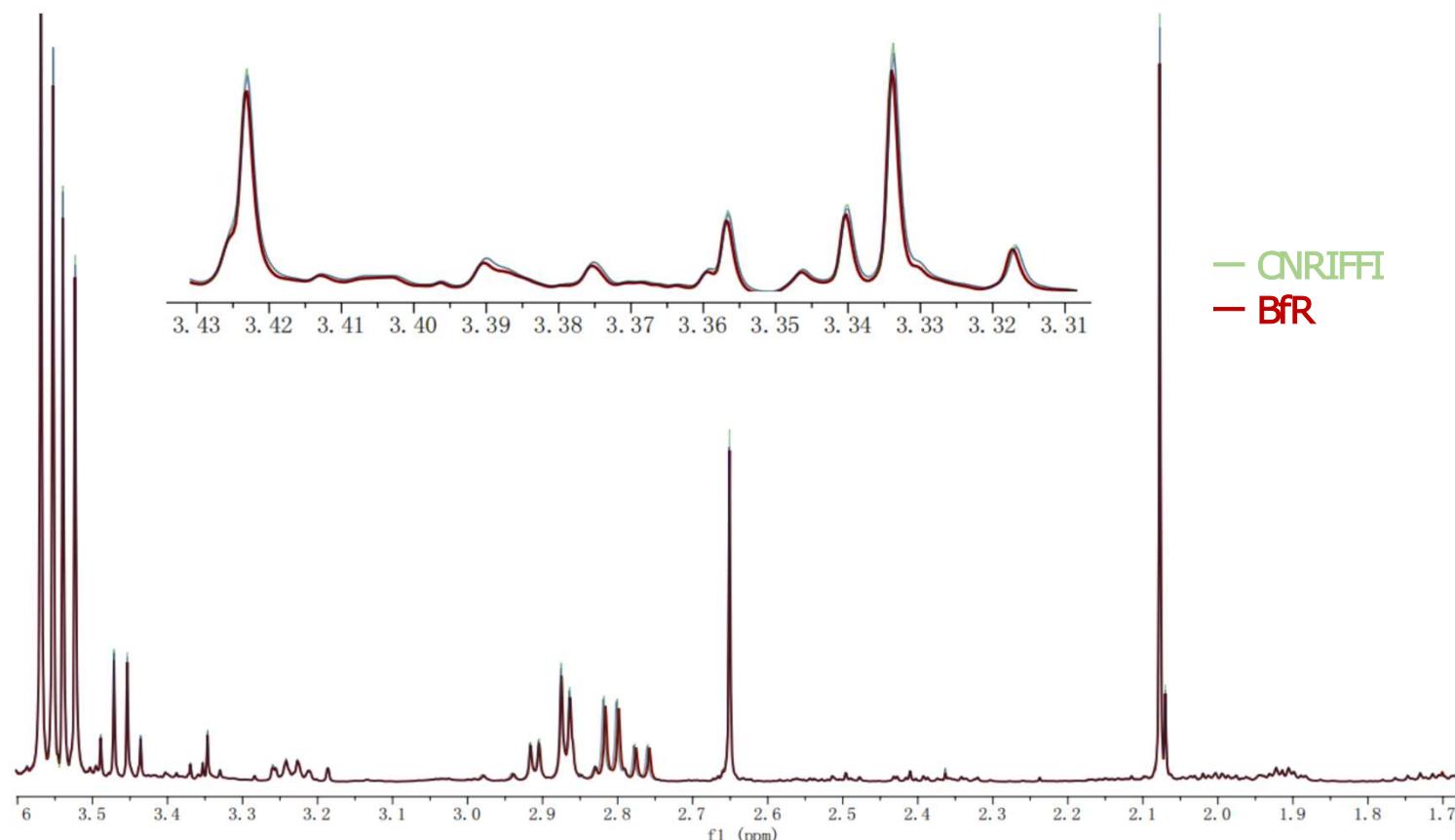
Sample	Institute	D/H I	D/H II	R
Ethanol	BfR	98.07	123.39	2.516
	NBCIQ	96.94	121.71	2.511
Ethanol	BfR	108.22	126.76	2.343
	NBCIQ	105.88	125.84	2.377
Baijiu	BfR	107.97	128.65	2.383
	NBCIQ	107.28	127.94	2.385
Baijiu	BfR	102.02	120.31	2.359
	NBCIQ	101.88	121.51	2.385
Baijiu	BfR	97.75	130.86	2.677
	NBCIQ	97.59	130.47	2.674
BfR Wine 1	BfR	100.63 (99.64-101.62)	126.75 (125-128.5)	2.519 (2.502-2.536)
	NBCIQ	101.59	128.22	2.524

➤ Wine Methods Equipment Innovation 葡萄酒分析设备创新

Development of ethanol distillation equipment for SNIF-NMR



➤ Wine Methods Verification Results 葡萄酒分析方法验证结果



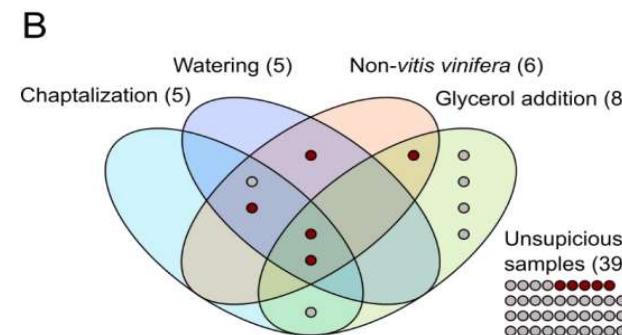
Note: Using the Same SOP of WineScreener 采用相同的WineScreener 标准操作流程

➤ Test results of 50 European labelling wines from EU-China-Safe project

Considering the stable isotope ratios of **D/H**, **18O/16O** and **13C/12C**, contents of technical glycerol by-products and anthocyanin composition, **more than 20 %** of the tested samples revealed anomalies.

- **watering (10%)**
- **chaptalisation(10%)**
- **technical glycerol addition(16%)**
- **non-vitis anthocyanin sources(12%)**

	Suspicious	Unsuspicuous
Based on labelling	10 (●)	40 (○)
Based on analysis results	11	39





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➤ EU-China-Safe Forums 中欧食品安全合作项目论坛





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➤ Awards 获奖



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