从技术层面探讨
——如何实现产品化学数据互认

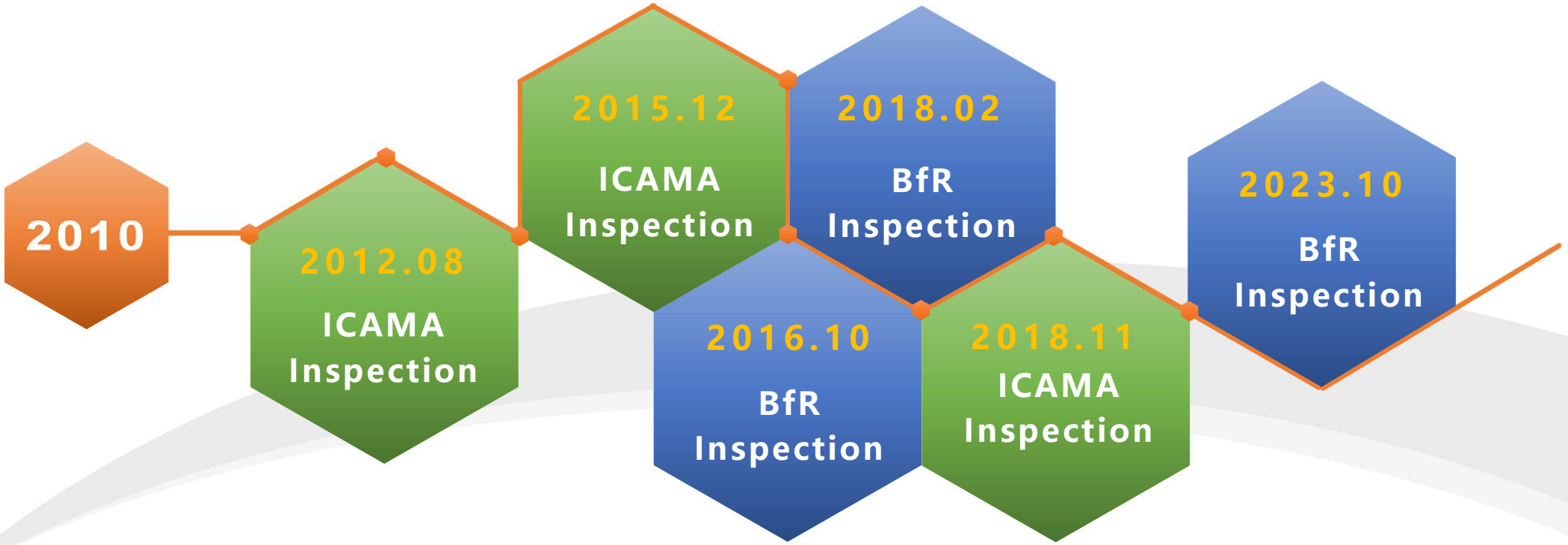
谢佩瑾

浙江省化工产品质量检验站有限公司
浙江省化工研究院化工产品检测中心

浙江省化工研究院化工产品检测中心

Test Center for Chemical Products of Zhejiang Chemical Industry Research Institute (ZCTC)

浙江省化工产品质量检验站有限公司
ZHEJIANG PROVINCE CHEMICAL PRODUCTS QUALITY INSPECTION STATION CO. ,LTD



浙江省化工研究院化工产品检测中心

Test Center for Chemical Products of Zhejiang Chemical Industry Research Institute (ZCTC)

Bundesinstitut für Risikobewertung
German Federal Institute for Risk Assessment
GLP-Bundesstelle / GLP Federal Bureau

BfR
Bundesinstitut für Risikobewertung

Gute Laborpraxis/Good Laboratory Practice

GLP-Bescheinigung/Statement of GLP Compliance
(gemäß/according to § 19b Abs. 1 Chemikaliengesetz)

Eine GLP-Inspektion zur Überwachung der Einhaltung der GLP-Grundsätze gemäß Chemikaliengesetz bzw. Richtlinie 2004/9/EG wurde durchgeführt in:

Assessment of conformity with GLP according to Chemicals Act and Directive 2004/9/EC at:

☒ Prüfeinrichtung/Test facility ☐ Prüfstandort/Test site

Test Center for Chemical Products of
Zhejiang Chemical Industry Research Institute
(ZCTC)

No. 387, Tianmushan Road
Hangzhou
Zhejiang Province - 310023, P. R. CHINA

(Unverwechselbare Bezeichnung und Adresse/Unequivocal name and address)

Prüfungen nach Kategorien/Areas of Expertise
(gemäß/according ChemVwV-GLP Nr. 5.3/OECD guidance)

1: Prüfungen zur Bestimmung der physikalisch-chemischen Eigenschaften und Gehaltsbestimmung

1: Physical-chemical testing

Datum der Inspektion/Date of Inspection
(Tag.Monat.Jahr/day.month.year)
25.10.2023 – 31.10.2023

Die/Der genannte Prüfeinrichtung/Prüfstandort befindet sich im nationalen GLP-Überwachungsverfahren und wird regelmäßig auf Einhaltung der GLP-Grundsätze überwacht.
Auf der Grundlage des Inspektionsberichtes wird hiermit bestätigt, dass in dieser Prüfeinrichtung/ diesem Prüfstandort die oben genannten Prüfungen unter Einhaltung der GLP-Grundsätze durchgeführt werden können.

The above-mentioned test facility/test site is included in the national GLP Compliance Programme and is inspected on a regular basis.
Based on the inspection report it can be confirmed, that this test facility/test site is able to conduct the aforementioned studies in compliance with the Principles of GLP.

Unterschrift, Datum / Signature, Date
Im Auftrag / By order

Berlin, 16.05.2024

Dr. Torsten Herold

Fachgruppenleitung „Krisenprävention und -koordination, Qualitäts- und Umweltmanagement, GLP-Bundesstelle" / Head of Unit "Crisis Prevention and Coordination, Quality and Environmental Management, GLP Federal Bureau"
(Name und Funktion der verantwortlichen Person / Name and function of responsible person)

Bundesinstitut für Risikobewertung, GLP-Bundesstelle, Max-Dohrn-Str. 8-10, 10589 Berlin, Germany
(Name und Adresse der GLP-Überwachungsbehörde / Name and address of the GLP Monitoring Authority)

农药登记试验单位证书

编号:	SD2024118
试验单位名称:	浙江省化工研究院化工产品检测中心
统一社会信用代码:	91330000717614261R
法定代表人（负责人）:	张建君
住所:	浙江省杭州市天目山路387号
实验室地址:	浙江省杭州市天目山路387号
试验范围:	产品化学试验：（全）组分分析试验、理化性质测定试验、产品质量检测试验/储存稳定性试验
有效期:	2024 年 05 月 20 日 至 2029 年 05 月 19 日

中华人民共和国农业农村部

2024 年 05 月 20 日

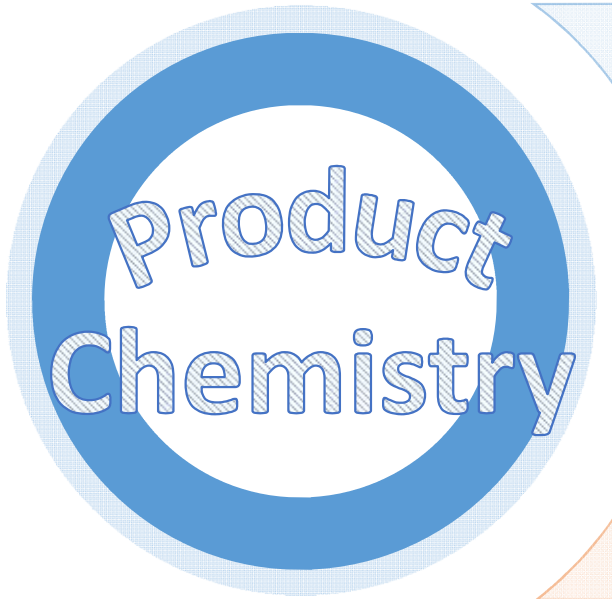
- Avoids duplication of testing
- Reduces use of animals
- Reduces trade barriers

Good Laboratory Practice (GLP)
Single quality standard for test facilities

Test Guidelines
Single quality standard for testing

Mutual Acceptance of Data (MAD)

Content



01

5-Bacthes Analysis

5-Batches Analysis

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- NY/T 2886-2016 农药登记原药全组分分析试验指南
- NY/T 2887-2016 农药产品质量分析方法确认指南

- COMMISSION REGULATION (EU) No 283/2013 1.11 Analytical profile of batches
- SANCO 3030/99 rev.5, 22 March 2019

- OPPTS 830.1700 Preliminary Analysis
- OPPTS 830.1800 Enforcement Analytical Method

- ABNT NBR 14029-2016 Pesticides – Validation of analytical methods

- APVMA Validation of analytical methods for active constituents and agricultural products

- CIPAC Guidelines on method validation to be performed in support on analytical methods for agrochemical formulations

5-Batches Analysis

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		NY/T 2886	EU 283	OPPTS 830.1700
Test substance		成熟定型 有代表性	Representative batches within the last five years of manufacture	5 batches (if batch production) 5 samples (if continuous production)
Impurities Screening				
Quantitative analysis		5 batches of active substances, additives, relevant impurities, significant impurities (≥1g/kg) Validation of analytical methods 980 g/kg ~1020 g/kg		
Qualitative analysis	**Active substances**	At least one batch UV/IR/NMR/MS **Counter ion**	At least one batch UV/IR/NMR/MS	
	Impurities	At least one batch One of UV/IR/NMR/MS	At least one batch **Highly specific methods** (i.e. mass spectrum)	
Others		Water	Water **Residual solvents**	

NY/T 2887-2016

如采用现行有效的**国家标准、行业标准、CIPAC和AOAC**标准方法，应进行特异性和定量限等必要参数的适应性试验。

SANCO/3030/99

Collaboratively tested standard **CIPAC, CEN, ISO and AOAC** methods for the analysis of the A.S. or impurities in technical active substance are regarded as validated. Therefore, no additional validation data are necessary, nevertheless the applicability of the method in terms of specificity must be demonstrated by example chromatograms. Where collaboratively tested **CIPAC or AOAC** methods are available, additional validation data are not required providing the method was collaboratively tested on the plant protection product type under consideration. However, the applicability of the method in terms of specificity must be demonstrated by example chromatograms (standard, blank : formulation without active substance, and formulation with active substance).

APVMA

The analytical methods for agricultural active constituents and agricultural chemical products described in the following documents are recognised as the **regulatory methods**:
Handbooks of the Collaborative International Pesticide Analytical Council (**CIPAC**)
The Association of Official Analytical Chemists' (**AOAC** International) Manual for agricultural active constituents and agricultral chemical product.

Method Validation

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Test Characteristics	Technical active substance		Plant protection product	
	Active substance	Significant impurity/ relevant impurities and additives	Active substance	Relevant impurities
Linearity	Yes	Yes	Yes	Yes
Accuracy (Recovery)	No	Yes	Yes	Yes
Precision	Yes	Yes	Yes	Yes
Range	Recommended	Yes	Yes	Yes
Limit of detection	No	Yes	No	Yes
Limit of quantitation	No	Yes	No	Yes
Interference	Yes	Yes	Yes	Yes
Specificity	Yes	Yes	Yes	Yes
Confirmatory of identity	No	Yes	No	Yes

Linearity

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Guideline	Technical active substance		Plant protection product	
	Active substance	Significant impurity/ relevant impurities and additives	Active substance	Relevant impurities
NY/T 2887	Range: 80%~120% 3 Levels ×2 or 5 Levels ×1 r≥0.99			
SANCO 3030	Range: 80%~120% 3 Levels ×2 or 5 Levels ×1 r≥0.99			
ABNT NBR 14029	Range: 80%~120% 5 Levels ×1 r≥0.99			
APVMA	Range: 80%~120% 3 Levels ×2 or 6 Levels ×1 r≥0.99			

APVMA

The specified range is normally derived from the linearity studies. The range of an analytical method is the interval between the upper and lower concentration (amounts) of analyte in the sample for which it has been demonstrated that the analytical method has suitable levels of precision, accuracy and linearity.

The following minimum specified ranges should be considered for the:

- assay of the active constituent of an agricultural chemical product, at least **80% to 120%** of the nominal concentration

- determination of an impurity, at least **from the specification level to 120%** of the specification level.

Precision (repeatability)

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Guideline	Technical active substance		Plant protection product	
	Active substance	Significant impurity/ relevant impurities and additives	Active substance	Relevant impurities
NY/T 2887	n≥5			
SANCO 3030	n≥5			
ABNT NBR 14029	**Repeatability assay 1:** same equipment, analyst, day (successive repetitions), n≥5 **Repeatability assay 2:** change one condition(equipment, analyst, day), n≥5 **Intermediate precision:** combine the data of repeatability assays 1 and 2			
APVMA	n≥5 Precision consider: **repeatability, intermediate precision and reproducibility**			

Precision (repeatability)

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Among-laboratory repeatability

- $\%RSD_R = 2^{(1-0.5\times\log(c))}$

Intra-laboratory repeatability

- $\%RSD_r = 0.67 \times 2^{(1-0.5\times\log(c))}$

Horrat value $H_r = \frac{\%\mathrm{RSD}}{\%\mathrm{RSD}_r}$

- $H_r \leq 1$ acceptance
- $1 < H_r \leq 2$ acceptance in case of a suggested explanation
- $H_r > 2$ not acceptance

Specificity/Selectivity

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- **Specificity** is the ability to **determine** the analyte in the presence of components
- **Selectivity** is the ability to **distinguish** between the analyte being measured and other substances

Specificity

refers to a method that produces a response for a single analyte only

Selectivity

refers to a method that provides responses for a number of chemical entities that may or may not be distinguished from each other

Interference

The specificity/selectivity is based on absence of interference.

Confirmatory of identity

Confirmatory techniques are required to support the identification of significant and relevant impurities, when the primary method of determination is not considered as highly specific.

Specificity/Selectivity

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Guideline	Technical active substance		Plant protection product	
	Active substance	Significant impurity/ relevant impurities and additives	Active substance	Relevant impurities
NY/T 2887	①通常采用光谱法，如GC/MS, LC/MS, HPLC-DAD ②干扰≤3% ③异构体拆分		非分析物的干扰：测定原药和空白样品	
SANCO 3030	Interference≤3% Enantiomer-selective		Formulation blank, standard, sample	
ABNT NBR 14029	Reading: blank reagent solution, blank sample Peak purity: DAD, MS, etc.			
APVMA	Peak purity: DAD, MS, etc.			
CIAPC	①Spectroscopic examination: GC/MS, LC/MS, HPLC-DAD ②Interference≤3% ③Non-analyte interference			

Specificity/Selectivity

Confirmation of identity is the unequivocal establishment of the **structural identity** of an analyte in a particular matrix based on **structural method** or by **comparison to standard material**, which could be characterized separately.

Non-specific method	Based on a functional group (moiety) within the analyte rather than for the specific analyte (ex: titration)	
Specific method	HPLC/GC	Retention time match
	HPLC-DAD	Retention time match + UV spectrum
Highly specific method	GC-MS or LC-MS	3 ions + retention time match
	LC-MS/MS	2 ions + retention time match
	Double HPLC-UV	Retention time match

LOD & LOQ

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LOD is the lowest amount of an analyte in a sample that **can be detected**, but not necessarily quantitated as an exact value.

LOQ is the lowest amount of the analyte in the sample that **can be quantitatively determined** with defined **precision** under the stated experimental conditions.

Guideline	Technical active substance		Plant protection product	
	Active substance	Significant impurity/ relevant impurities and additives	Active substance	Relevant impurities
NY/T 2887	No	< 0.1%	No	
SANCO 3030	No	Specification (20% below) Standard addition (recovery+precision)	No	Specification
ABNT NBR 14029	DL is defined as lowest concentration that may safely be detected (e.g. **S/R=3**) QL is defined as **first level concentration of the linear range** (%RSD < %RSDr(n=5),S/R≥10)			
APVMA	LOD = X + (3 × SD) LOQ = X + (10 × SD)			

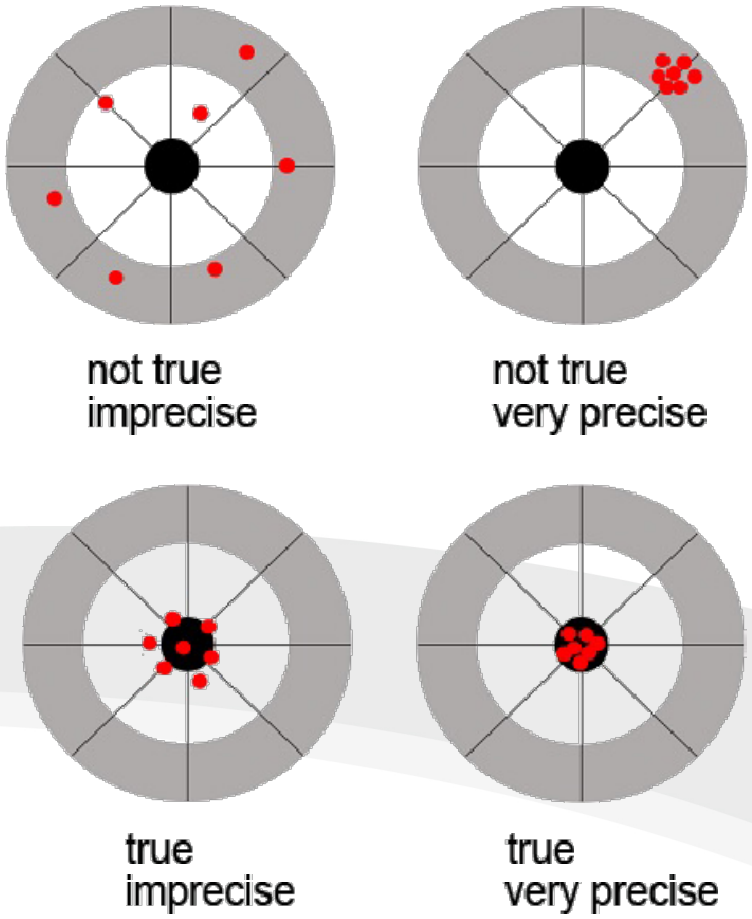
Accuracy

Accuracy

Trueness
(recovery)

+

Precision
(repeatability)



Recovery

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Guideline	Technical active substance		Plant protection product	
	Active substance	Significant impurity/ relevant impurities and additives	Active substance	Relevant impurities
NY/T 2887	No	标准品添加法 1 level×5 (1:1) 3 levels×2 (0.8:1, 1:1, 1:1.2)	实验室合成样品/标准品添加法 1 level×4	同原药中杂质
SANCO 3030	No	①Methods ∝ matrix ②Levels ∝ specification ③2 independent determinations ④Mean recovery + RSD (n > 2)	①Lab-prepared sample/standard addition ②(90%~110%)X ③2 independent determinations ④Mean recovery + RSD (n > 2)	recovery and precision can be determined by fortification of blank formulations.
ABNT NBR 14029	Standard addition or lab-prepared sample 3 levels: 80%, 100%, 120%			
APVMA	Standard addition or lab-prepared sample 3 levels: 80%, 100%, 120%			

Recovery

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1. **Total recovery** based on **the native plus added analyte**
2. **Marginal recovery** based on **the added analyte**

- Total recovery

$C_U < 10\%C_A$

$$\text{Total \%recovery} = 100 \times \frac{C_F}{C_U + C_A}$$

- Marginal recovery

$C_U > 10\%C_A$

$$\text{Total \%recovery} = 100 \times \frac{C_F - C_U}{C_A}$$

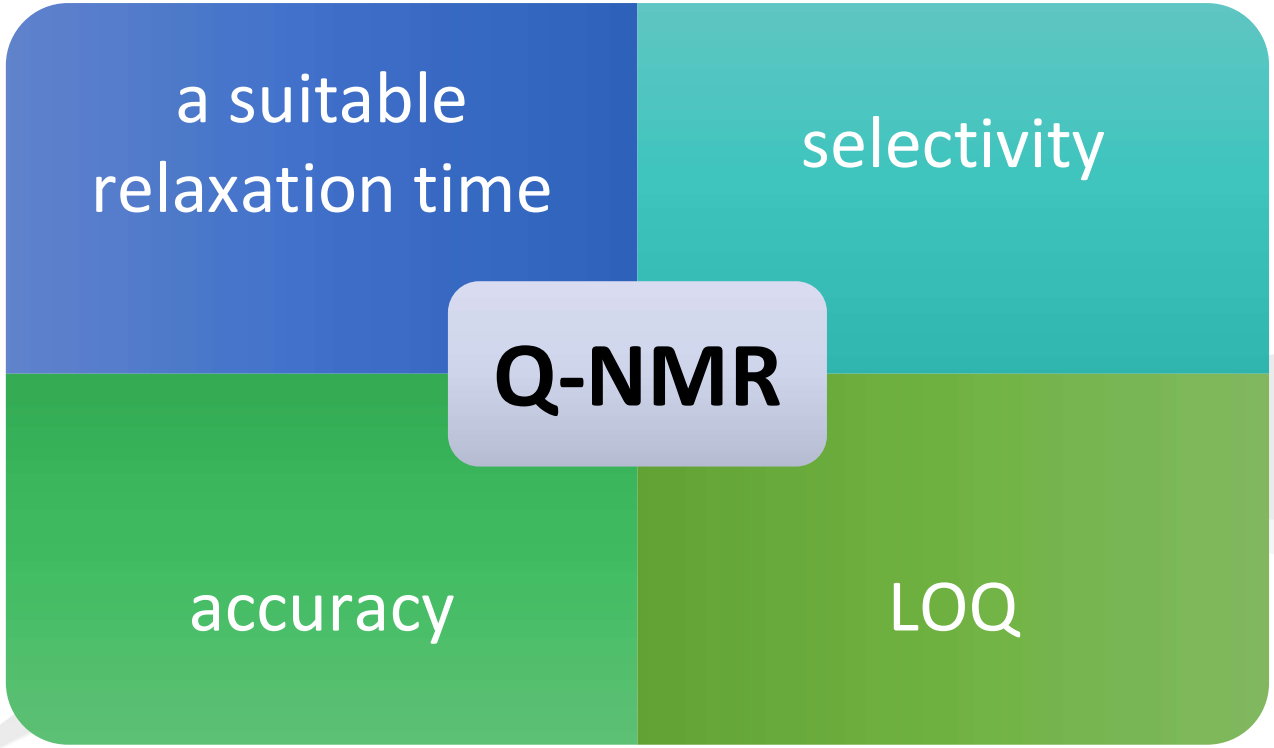
Q&A

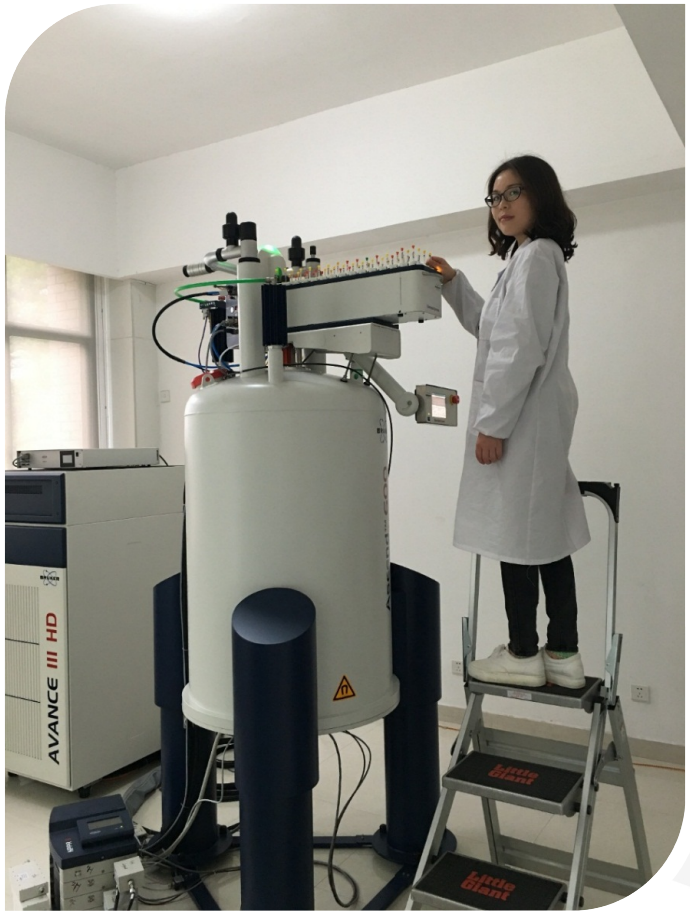
How to test the recovery for undetected relevant impurities?

- **Specification ?**
- **LOQ ?**
- **Precision?**

Method validation of Q-NMR

APVMA 5.3. Typical characteristics for quantitative Nuclear Magnetic Resonance (NMR) data





02

Physical and chemical properties

Physical and chemical properties

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- **NY/T 1860** 农药理化性质测定试验导则（第1部分~第38部分）
- **OECD Guidelines** for the Testing of Chemicals
- Commission Regulation (EU) **No 283**/2013 Data requirements for active substances
- Commission Regulation (EU) **No 284**/2013 Data requirements for plant protection products
- Council Regulation (EC) **No 440**/2008 Laying down test methods pursuant to Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH)
- EPA 40 CFR **Part 158** Data Requirements for Pesticide Registration
- EPA OPPT **Series 830** Product Properties Test Guidelines
- Manual of Tests and Criteria, United Nations Recommendations on the Transport of Dangerous goods.

Categories of test substance

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ICAMA 2569	Technical materials	Active ingredient （AI, > 98%）
		Technical materials as manufactured(TC)
	Formulations	
EC 1107 EU 283 EU 284	Active substances	Purified active substances(PAS)
		Active substances as manufactured(MAS)
	Plant protection product	
OPPTS 830.1000	Active ingredients	Pure active ingredients (PAI)
		Technical grade of active ingredients (TGAI)
	Manufacturing-use product (MP)	
	End-use product (EP)	

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Data requirements of physical and chemical properties

Physical and chemical properties of **active substance**

Properties	ICAMA 2569	EU 283	40 CFR Part 158
Appearance	**AI+TC**	PAS+MAS	TGAI+MP
Melting point/range	**AI+TC**	PAS	TGAI or PAI (solid)
Boiling point	**AI+TC**	PAS	TGAI or PAI (liquid)
Solubility in water	AI	PAS	TGAI or PAI
Solubility in organic solvents	AI	PAS or MAS	
Density/relative density/bulk density	AI		TGAI+MP
Partition coefficient (n-octanol/water)	AI	PAS	TGAI or PAI
Vapor pressure	AI	PAS & **Volatility**	TGAI or PAI
Dissociation Constants	AI	PAS	TGAI or PAI
Hydrolysis	AI	environment	environment
Photolysis	AI	environment	environment
UV/visible light absorption	AI	**UV/VIS, IR, NMR, MS, molar extinction at relevant wavelengths, optical purity**	TGAI or PAI
Specific optical rotation	AI+TC		
Surface tension		PAS	

Physical and chemical properties of **active substances**

Properties	ICAMA 2569	EU 283	40 CFR Part 158
Particle size, fiber length, and diameter distribution			TGAI or PAI
Stability to normal and elevated temperatures, metals, and metal ions	TC		MP + TGAI
Explodability	TC	MAS	MP
Flammability	TC	MAS Flammability and self-heating Flash point (m.p. < 40℃)	MP
Oxidation/reduction: chemical incompatibility	TC	MAS Oxidising properties	MP
Corrosion characteristics	Packaging material		MP
Storage stability			MP
Miscibility			MP
pH			MP + TGAI
Viscosity			MP (liquid)
Density			MP + TGAI

Physical and chemical properties of **the plant protection product**

Properties	ICAMA 2569	EU 283	40 CFR Part 158
Appearance	all	all	EP
Density/relative density/bulk density	all	relative density: liquid bulk density: solid	EP, MP, TGAI
Oxidation/reduction: chemical incompatibility	all	**oxidising properties**	EP, MP
Corrosion characteristics	all		EP, MP
Explodability	all	all	EP, MP
Flammability of solids	solid	solid (self-heating)	EP, MP
Flash point	liquid	liquid	
Viscosity	liquid	liquid (surface tension)	EP, MP
Miscibility	OL		
Acidity/alkalinity and pH value		all	EP, MP
Stability to normal and elevated temperatures, metals, and metal ions			EP, MP, TGAI
Storage stability and shelf-life			EP, MP
Dielectric breakdown voltage			EP

Data requirements of physical and chemical properties

Properties	ICAMA 2569	EU 283	40 CFR Part 158
Vapor pressure	not required for salts	① 20℃ or 25℃ ② volatility (Henry's law constant) for solids and liquids	not required for salts
Dissociation constants in water	weak acid, weak base	① identity of the dissociated species formed ② determine the non-dissociated form for salt	contains an acid or base functionality (organic or inorganic) or an alcoholic functionality (organic)
Partition coefficient (n-octanol/water)	organic and non-polar	① 20℃ or 25℃ ② if 2≤pKa≤12, the effect of pH (4 to 10) shall be investigated	organic and non-polar
Solubility in water		① in the neutral range ② if 2≤pKa≤12, that shall be determined in the acidic range (pH 4 to 5) and in the alkaline range (pH 9 to 10)	
Solubility in organic solvents	polar, non-polar, aromatic	① aliphatic hydrocarbon (heptane) ② aromatic hydrocarbon (toluene) ③ halogenated hydrocarbon (dichloromethane) ④ alcohol (methanol/isopropyl alcohol) ⑤ ketone (acetone) ⑥ ester (ethyl acetate)	not required

02-1

Test methods of physical and chemical properties

Test methods of physical and chemical properties

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Properties		ICAMA NY/T	OECD TG	EPA OPPTS	Other
pH		1860.1	122	830.7000	CIPAC MT 75
Acidity and alkalinity		1860.2		/	CIPAC MT 31 CIPAC MT 191
Appearance	Color	1860.3	/	830.6302	
	Physical state			830.6303	
	Odor			830.6304	
Thermal Stability		1860.4	113		
UV-VIS absorption		1860.5	101	830.7050	
Explodability		1860.6		830.6316	EC 440 A.14 UN Manual
Partition coefficient (n-octanol/water)	Shake flask method	1860.8	107 (-2~4)	830.7550	EC 440 A.8
	Generator column method			830.7560 (1~6)	
	Estimation by liquid chromatography		117(0~6)	830.7570	
	Slow-stirring method		123(~8.2)		

Test methods of physical and chemical properties

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Properties		ICAMA NY/T	OECD TG	EPA OPPTS	Other
Hydrolysis		1860.9	111	796.3500	
Oxidation/reduction: chemical incompatibility		1860.10	/	830.6314	
Flash point		1860.11	/	830.6315	EC 440 A.9 ASTM D 1310-01
Fire point		1860.12	/		ASTM D 1310-01 ISO 2592
Miscibility		1860.13	/	830.6319	CIPAC MT23
Vapor pressure		1860.14	104	830.7950	EC 440 A.4
Flammability	Solids	1860.15	/	830.6315	EC 440 A.10
	Gases	1860.25			EC 440 A.11
	Aerosols	1860.27			UN Manual
	Contact with water	1860.29			EC 440 A.12 UN Manual
Relative self-ignition temperature for solids		1860.24	/		EC 440 A.16
Auto-ignition temperature (liquids and gases)		1860.26	/		EC 440 A.15

Test methods of physical and chemical properties

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Properties	ICAMA NY/T	OECD TG	EPA OPPTS	Other
Corrosion characteristics	1860.16	/	830.6320	ASTM G31-72 UN Manual
Density/relative density/bulk density	1860.17	109	830.7300	CIPAC MT 3 CIPAC MT 33 CIPAC MT 159 CIPAC MT 169 CIPAC MT 186 EC 440 A.3
Specific optical rotation	1860.18			ISO 6353-1
Boiling point/boiling range	1860.19	103	830.7220	EC 440 A.2
Melting point/melting range	1860.20	102	830.7200	EC 440 A.1
Viscosity	1860.21	114	830.7100	CIPAC MT 22
Solution on organic solvents	1860.22	/	/	CIPAC MT 181

Test methods of physical and chemical properties

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Properties	ICAMA NY/T	OECD TG	EPA OPPTS	Other
Water solubility	1860.23	105	830.7840 830.7860	EC 440 A.6
Oxidation	1860.28			EC 440 A.17 (Solids) EC 440 A.21 (Liquids)
Dissociation Constants	1860.30	112	830.7370	
Surface Tension of Aqueous Solutions	1860.31	115		EC 440 A.5
Particle Size Distribution-Fibre Length and Diameter	1860.32	110		
Stability to normal and elevated temperatures, metals and metal ions	1860.38		830.6313	

Appearance-Color

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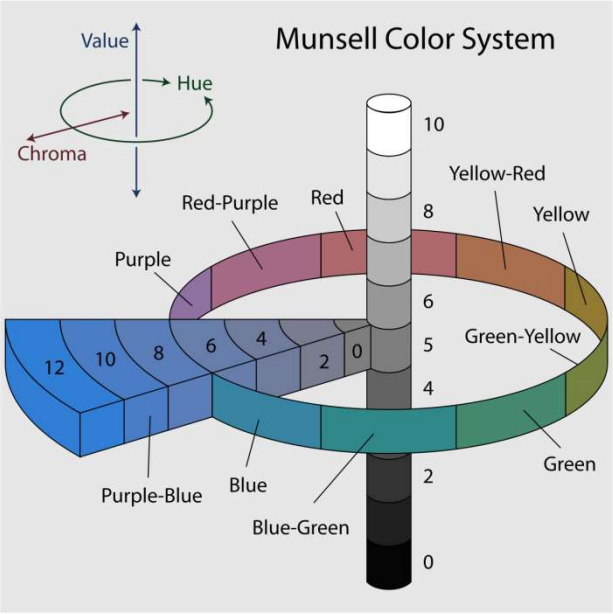
OPPTS 830.6302 Color

a) Visual （qualitative）

b) ASTM D-1535 Standard Method of Specifying Color by the Munsell System

c) ASTM D-1544 Standard Test Method for Color of Transparent Liquids (Gardner Color Scale)



Corrosion characteristics

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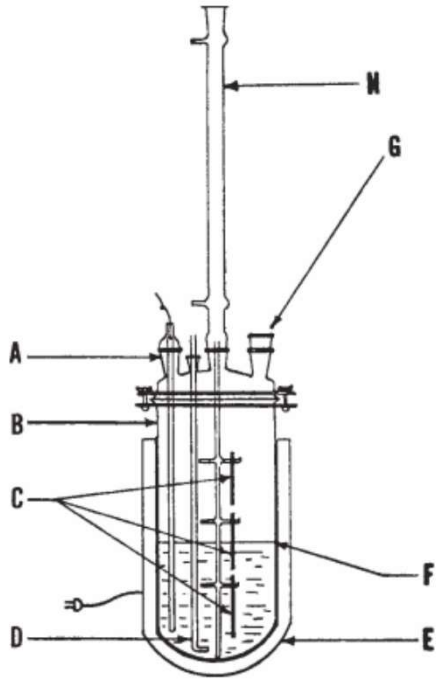
Corrosion characteristics: is to evaluate effects of the product formulation on its **packaging material**.

OPPTS 830.6320 Corrosion Characteristics

- Corrosion characteristics to packaging material OPPTS 830.6317 (Storage stability)
- Immersion Corrosion Testing of Metals ASTM G31-72(2004)

OPPTS 830.6313 Stability to Normal and Elevated Temperature, Metals, and Metal Ions



NOTE 1—The flask can be used as a versatile and convenient apparatus to conduct simple immersion tests. Configuration of top to flask is such that more sophisticated apparatus can be added as required by the specific test being conducted. *A* = thermowell, *B* = resin flask, *C* = specimens hung on supporting device, *D* = air inlet, *E* = heating mantle, *F* = liquid interface, *G* = opening in flask for additional apparatus that may be required, and *H* = reflux condenser.

FIG. 1 Typical Resin Flask

Water solubility

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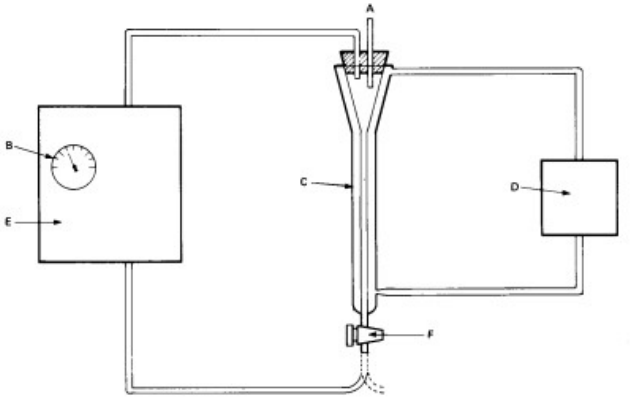
Preliminary Test

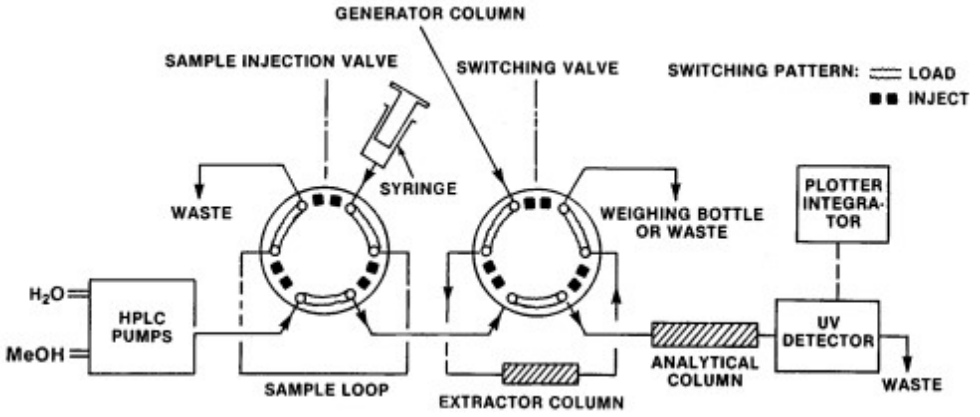
Column elution method
(< 10^{-2} g/L)

Flask method
(> 10^{-2} g/L)

Generator column method
(1ppb~5000ppm)



A. Atmospheric equilibration
B. Flowmeter
C. Microcolumn
D. Thermostatically controlled circulating pump
E. Recirculating pump
F. Two-way valve for sampling



EC 283 § 4.1.2 Methods for risk assessment

(g) in water, buffer solutions, organic solvents and any additional matrices used **in the physical and chemical properties tests**.

The **specificity** of the methods shall be determined and reported. Validated confirmatory methods shall be submitted if appropriate.

The **linearity, recovery and precision (repeatability)** of methods shall be determined and reported.

Data shall be generated at the **LOQ** and either the likely residue levels or ten times the LOQ. Where relevant, the LOQ shall be determined and reported for each analyte.

Test methods of storage stability

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Properties	ICAMA	EPA	CIPAC	Others
Stability at elevated temperature	GB/T 19136	830.6317 (40℃~54℃)	MT 46.4	OECD 113
Stability at 0℃ (for liquid)	GB/T 19137	830.6317 (-20℃~0℃)	MT 39.3	FAO 4.6.1
Freeze/thaw stability (for CS)	GB/T 43273			FAO 7.33
Shelf life (at ambient temperature)	NY/T 1427	830.6317		Croplife No.17

GB/T 19136-2003

Temperature	Period
54℃±2℃	2 weeks

GB/T 19136-2021

Temperature	Period
54℃±2℃	14 days
50℃±2℃	4 weeks
45℃±2℃	6 weeks
40℃±2℃	8 weeks
35℃±2℃	12 weeks
30℃±2℃	18 weeks

Test methods of shelf life

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	ICAMA	EU	EPA
Guideline	NY/T 1427	EU 284	830.6317
Test item	Formulation	Plant Protection Products	TGAI, MP, EP
Condition	≥20℃, (30±2)℃ is recommend Humidity, light (if any)	Ambient temperature	T: 20℃ or 25℃ H: 50% (if any)
Period	2 Y	2 Y	≥ 1Y
Test interval	0, 3M, 6M, 12M, 24M (n≥5)	In months	0, 3M, 6M, end
Package	Commercial package or in smaller packages of the same construction and materials	Commercial package	Commercial package or in smaller packages of the same construction and materials
Results	Appearance, weight, content (ai, impurity, additives), technical characteristics	Appearance, content (ai, impurity), technical characteristics	Appearance, weight, content (ai, impurity), physical changes

Test methods of technical characteristics

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Category	Properties	ICAMA	CIPAC	Formulation	Requirement
Relevant impurity	Water	GB/T 1600-2021	MT 30.2 MT 30.6	TC, TK, EC, UL, DC, OL, OD…	
	Loss in weight	GB/T 30361-2013	MT 17		
	Insolubles	GB/T 19138	MT 10 Water	TC, TK	
		GB/T 28136	MT 27 Acetone		
		GB/T 43179(DMF)	MT 7 Ethanol MT 71 NaOH MT 11 Xylene MT 35 Oil		
Density	Relative density	GB/T 32776	MT 3		
	Bulk density	NY/T 1860.17 GB/T 33810	MT 186	GR	
Surface	Wettability	GB/T 5451	MT 53.3	solid to be dispersed or dissolved in water	< 1min
	Persistent foaming	GB/T 28137	MT 47.3	to be diluted with water	< 60mL (1min)

Test methods of technical characteristics

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Category	Properties	ICAMA	CIPAC	Formulation	Requirement
Particulate, fragmentation and adhesion	Wet sieve test	GB/T 16150	MT 182 MT 185 MT 59.3 MT 167	WP, SC, FS, OD, WG, WS, CS, DC, SE, ST, WT, EG, EP, ZC, ZW, ZE	≤2% （75μm）
	Dry sieve test	GB/T 16150	MT 170	DP, DS	
	Size range	HG/T 2467.12	MT 170 MT 187	GR	≥85%
	Dustiness	GB/T 30360	MT 171.1	GR, WG, EG, SG	≤ 30mg ≤ 25 （Optical）
	Attrition resistance	HG/T 2467.12 GB/T 33031	MT 178 MT 178.2 (< 1cm)	GR, WG, SG, EG, DT, WT, ST	
	Tablet integrity	visual	visual	DT, ST, WT	
	Tablet hardness		MT 193		
	Adhesion to seeds	GB/T 17768 GB/T 43174	MT 194	Seed treatment	

Test methods of technical characteristics

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Category	Properties	ICAMA	CIPAC	Formulation	Requirement
Volatilization	Volatility			UL	
Dispersion	Dispersibility and spontaneity of dispersion	HG/T 2467.13	MT 160 MT 174	SC, CS, ZC, WG	≥60%
	Disintegration time and dispersibility/dissolution	GB/T 32775	MT 196 MT 197	ST, WT	
	Suspensibility	GB/T 14825	MT 184.1	CS, FS, SC, ZC, WG, WP, WT	60%~105%
	Dispersibility	GB/T 32775	MT 174		
	Dispersion stability	HG/T 2467.11	MT 180	SE, EG, EP, DC, OD, ZW, ZE	
	Emulsion stability and re-emulsification	GB/T 1603	MT 36.3	EC, EW, ES, ME	

Test methods of technical characteristics

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Category	Properties	ICAMA	CIPAC	Formulation	Requirement
Flow	Flowability	GB/T 34775	MT 172.2		
	Pourability	GB/T 31737	MT 148.1		
	Viscosity	NY/T 1860.21	MT 192 MT 22		
Solution and dissolution	pH	GB/T 1601	MT 75.3		
	Acidity and alkalinity	GB/T 28135	MT 31 MT 191		
	Miscibility with hydrocarbon oil	/	MT 23		
	Dissolution of water soluble bags	/	MT 176		
	Degree of dissolution and/or solution stability	HG/T 2467.15 GB/T 32777	MT 179.1 MT 41.1 MT 196		
	Dissolution of water soluble bags	/	MT 176	Dissolution of water soluble bags	

New decisions of CIPAC Methods

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MT 30.6	The revision of the MT 30.5, Karl Fischer method using pyridine-free reagents (CIPAC/5154) was accepted as full CIPAC method.
MT 172.2	The revision of the CIPAC MT 172.1, Flowability of Granular Formulations (CIPAC/5155) was accepted as full CIPAC method
MT 184.1	The revision of the CIPAC MT 184, Suspensibility of formulations forming suspensions on dilution with water (CIPAC/5156) was accepted as full CIPAC method.
MT 46.4	The harmonized accelerated storage procedure for all formulation types (CIPAC/5217) was accepted as a full CIPAC method. MT 46.4 supersedes all previous versions of MT 46 for accelerated storage.
MT 178.3 attrition resistance	The revision of methods MT 178 and MT 178.2 (CIPAC/5321) to combine into a single method for granular products and to include loosely packed tablets was accepted as full CIPAC method with the editorial changes and with the remark that MT 178.3 supersedes MT 178 and MT 178.2.

New decisions of CIPAC Methods

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MT 201 Discharge rate of trigger dispenser	The method for determination of the discharge rate of trigger dispensers (CIPAC/5152) was accepted as full CIPAC method as Technical monograph No.2 (8th edition) contains the formulation type TD (trigger dispenser)
MT 202 Discharge rate of aerosol dispenser	The method for determination of the discharge rate of aerosol dispenser (CIPAC/5153) was accepted as full CIPAC method.
MT 160.1 Spontaneity of dispersion of suspension concentrates	The revision of methods MT 160 (CIPAC/5323) to determine the spontaneity of dispersion of liquid formulations forming suspensions on dilution with water was accepted as full CIPAC method with the remark that MT 160.1 supersedes MT 160.

New decisions of CIPAC Methods

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MT 185.1 Wet sieve test	The revision of methods MT 182 and MT 185 (CIPAC/5353) to combine into a single method for wet sieve test was accepted as full CIPAC method under the prerequisite that it supersedes both MT 182 and MT 185
MT 148.2 Pourability	The revision of methods MT 148 and MT 148.1 (CIPAC/5355) to combine into a single method MT 148.2 for the determination of pourability of formulations was accepted as provisional CIPAC method with the remark that MT 148.2 supersedes MT 148 and MT 148.1.
MT XXX Density of solids and liquids with automated systems	The method intended to determine the density of solids and liquids (CIPAC/5356) with automated systems with two different procedures (gas pycnometer for solids, oscillating density meter for liquids) was accepted as provisional CIPAC method

Summary

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ZHEJIANG PROVINCE CHEMICAL PRODUCTS QUALITY INSPECTION STATION CO. ,LTD

Thank You for Your Attention