涂料中铅的测定方法与质控措施

Analytical Methods and Quality Assurance Strategies for Lead in Paint

环境保护部国家环境分析测试中心

National Research Center for Environmental Analysis and Measurement

2016.4.26

主要内容 Outline

01 中心简介 Introduction of NERC

02 分析方法 Methodology

03 质控措施 Quality Assurance

04 比对实验室简介
Lab for Intercomparison

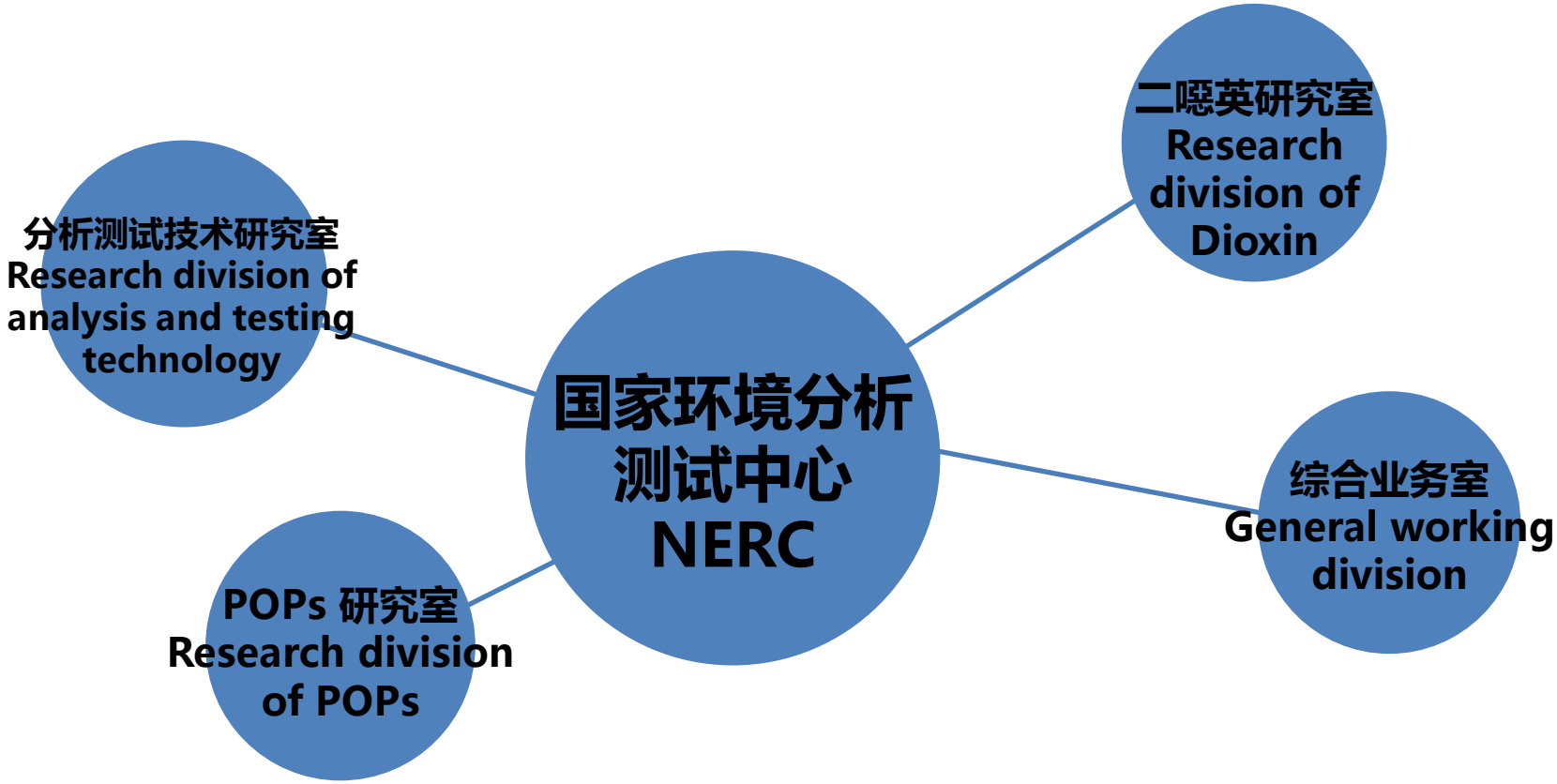
中心简介 Introduction of NERC

- 环境保护部直属单位（副局级），成立于1984年
- 科技部所属国家分析测试中心之一
- 环境保护部环境管理和决策的技术支持单位
- 环境保护部在环境科学领域开展国内外环境问题合作研究和交流的窗口
- 2008年“国家环境保护二恶英污染控制重点实验室”挂牌
- NERC, department of MEP, was founded in 1984,also one of the national analysis and Testing Center of Ministry of science and technology. NERC is technical support unit for environmental management and decision making of MEP. NERC carry out the research of environmental science and the development of domestic and international cooperation."Key Laboratory of dioxin pollution control of MEP" were listed in 2008.

中心简介 Introduction of NERC

- 配合环境保护部的管理需求，开展环境分析测试新技术的研究，从事环境科学综合性分析测试技术和方法研究
  Study on the new technology of environmental analysis and testing, study on the technology and method of comprehensive analysis and testing of environmental science
- 着重解决我国环境分析测试领域中的关键性和综合性问题
  Solve the key problems in the field of environmental analysis and testing in China
- 开发、引进和推广环境分析测试的新技术、新产品
  Development, introduction and promotion of new technologies and products for environmental analysis and testing
- 承担污染纠纷的仲裁性分析测试任务
  Undertake the arbitration analysis and testing task of pollution dispute
- 围绕全球性重点环境问题开展国内外合作研究和技术交流
  Focus on global environmental issues and carry out cooperative research and technical exchanges nationally and international ly

中心简介 Introduction of NERC



中心共有在职职工46人，其中研究员6人，副研究员和高级工程师10人，工程师12人；5人为归国留学人员，13人取得博士学位。

46 employees of all in the center, 6 senior researchers, 10 deputy researchers or senior engineers, 12 engineers, 5 oversea returnees, 13 persons with the doctor's degree.

管理支撑 Management Support

全国医疗废物和危险废物二噁英验收监测
National inspection and monitoring on Dioxin of medical waste and hazardous waste

全国汞污染防治管理
National mercury pollution prevention and control management

持久性有机污染物监测
Monitoring of persistent organic pollutants

全国重点地区环境与健康专项调查
Special investigation on the environment and health in the key areas of China

全国土壤污染状况调查及污染防治
Investigation and pollution control of soil pollution in China

国家环境分析测试中心作为环境保护部下属单位，积极承担国家环境管理专项任务，努力为环境管理提供技术支撑服务。

NERC, as a subordinate unit of MEP, undertake the special task of national environmental management, and provide technical support services for environmental management.

科研成果 Scientific Research Achievements

研究人员在沙尘暴源区采集样品
Sampling in source areas of dust storms

环境保护科学技术奖

获奖证书

获奖项目：沙尘暴与黄沙对北京地区大气颗粒物影响研究

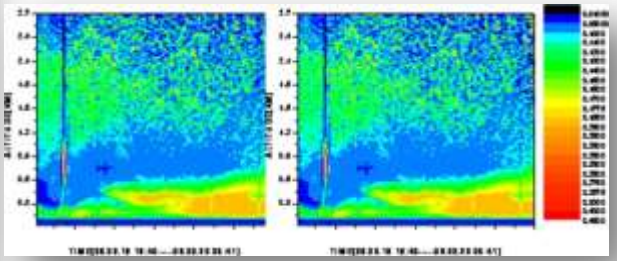
获奖等级：三等

获奖单位：国家环境分析测试中心（第一完成单位）

二〇〇三年十月

证书号：KJ2003-3-18-D01

北京沙尘暴三大源区示意图
Three main source areas of dust storms in Beijing



激光雷达观测图
Observation of laser radar

沙尘暴与黄沙对北京地区大气颗粒物影响研究

Study on the influence of dust storm to atmospheric particles in Beijing

科研成果 Scientific Research Achievements

热光碳质组分测定仪
Thermal/optical carbon analyzer

戴安离子色谱仪
Diane ion chromatography

安捷伦7700xICP-MS无机质谱仪
Agilent ICP-MS 7700x

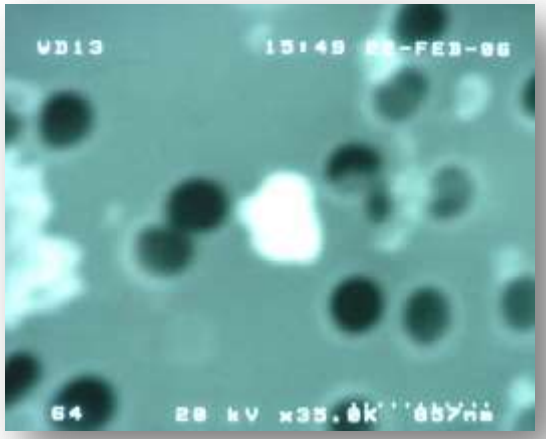
日立S2700扫描电镜-能谱仪
Hitachi S2700 of SEM-EDS


二 等 奖

项 目 名 称 ：

报 送 单 位 ：

参 加 人 员 ：

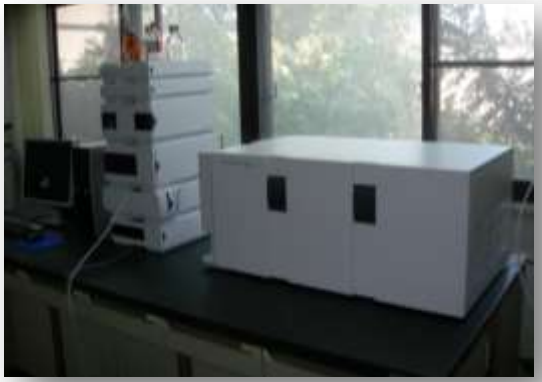


含铅颗粒
Particle with Lead

大气颗粒物分析表征及来源解析

Characterization and source apportionment of atmospheric particulate matter

科研成果 Scientific Research Achievements



液相色谱/三重四级杆质谱
HPLC/MS/MS

气相色谱/(NCI)四级杆质谱
GC/NCI

TD-GC×GC/q-MS

HPLC/MS

环渤海沿岸OCPs的残留
Residues of OCPs along the coast of Bohai

环境持久性有机污染物研究
Study on environmental POPs

科研成果 Scientific Research Achievements

制订国家环境保护技术标准
Formulating national standards for environmental protection

环境二噁英类飞灰标准样品研究
Study on fly ash references of dioxin

二噁英类污染物研究
Study on Dioxin

分析方法 Analysis Method

可溶性铅 Soluble Lead

室内装饰装修材料 内墙涂料中有害物质限值

Indoor decorating and refurbishing materials – Limit of harmful substances of interior architectural coatings

GB 18582-2008

总铅 Total Lead

建筑用外墙涂料中有害物质限值

Limit of harmful substances of exterior wall coatings

GB 24408-2009

可溶性铅 Soluble Lead

涂膜的制备 Preparation of the coating	样品处理 Sample preparation	测试 Testing
样品混匀后在玻璃板上制备涂膜，干燥后取下涂膜，粉碎过筛。 Coating, drying, grinding, sieve.	取0.5g试样，加入25ml，0.07 mol/l 盐酸，搅拌1h，放置1h，过滤后待测。 Take the 0.5g sample, add 25ml, 0.07 mol/l hydrochloric acid, stir 1h, place 1h, after filtering to be measured.	原子吸收光谱仪测定 Atomic absorption spectrometry

GB 18582-2008 附录D

总铅 Total Lead

涂膜的制备 Preparation of the coating	样品处理 Sample preparation	测试 Testing
样品混匀后在玻璃板上制备涂膜，干燥后取下涂膜，粉碎过筛。 Coating, drying, grinding, sieve.	1. 干灰化法 Dry ashing 2. 湿酸消解法 Wet acid digestion 3. 微波消解法 Microwave digestion	原子吸收光谱仪 Atomic absorption spectrometry ICP-OES ICP-MS

GB 24408-2009 附录E

样品处理 Sample Preparation

干灰化法 Dry ashing

取0.2-0.3g样品放入坩埚内，0.5g $MgCO_3$覆盖在样品上，电热板加热475℃至碳质残渣，硝酸复溶，过滤后定容待测。

Take 0.2-0.3g sample into the crucible, 0.5g of $MgCO_3$ covered in samples, electric heating plate 475℃ to carbonaceous residue, nitrate complex solution, filtered to be measured.

湿酸消解法 Wet acid digestion

取0.1-0.3g样品放入烧杯内，7mL 硝酸，电热板加热至微沸，溶液冒白烟后加入1-2mL 双氧水，至样品完全消解后稀释定容待测。

0.1-0.3g samples were placed in a beaker, 7ml nitric acid, electric heating plate to micro boiling. When white smoke appeared, add hydrogen peroxide 1-2mL. After complete digestion, dilution will be measured.

微波消解法Microwave digestion

取0.1-0.2g样品放入消解罐内，5mL硝酸+2mL双氧水，梯度升温至180℃消解10min，降温后稀释定容待测。

0.1-0.2g sample in the digestion tank, 5mL +2mL nitrate hydrogen peroxide, gradient is heated to 180 ℃ for 10min digestion, dilution, measured after cooling.

GB 24408-2009 附录E

质控措施 Quality Assurance

全程序空白 Full procedure blank sample

每10个样品1个全程序空白，不足10个样品含1个全程序空白。
1 full procedure blank sample for each of the 10 samples, 1 full procedure blank sample, when less than 10 samples .

质控样品 QC sample

每10个样品1个涂料质控样品，不足10个样品含1个质控样品。
1 QC sample for each of the 10 samples, 1 QC sample, when less than 10 samples .

平行样品 Parallel sample

每10个样品1个平行样品，不足10个样品含1个平行样品。
1 parallel sample for each of the 10 samples, 1 parallel sample, when less than 10 samples .

再现性 Repeatability

不同实验室间测试结果的相对偏差应小于20%。
The relative deviation of the test results in different laboratories should be less than 20%.

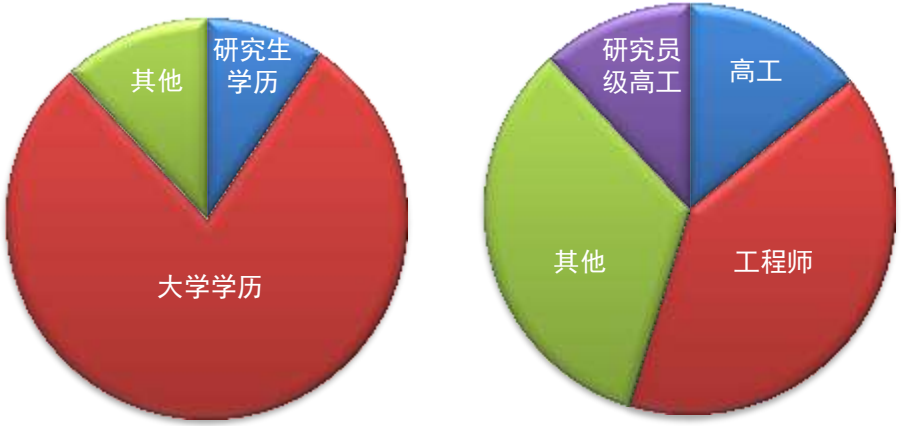
国家涂料质量监督检验中心
National Quality Supervision Testing Center for Paint

1982	•化工部涂料和颜料监测中心授权 Authorized by the Ministry of chemical industry paint and pigment monitoring center
1988	•国家涂料质量监督检验中心授权 Authorized by national paint quality supervision and inspection cente
1990	•首次通过计量认证 China Metrology Accreditation
1998	•首次通过实验室认可 Laboratory accreditation assessment by China National Accreditation Service for Conformity assessment

组织构成 Organization structure

- 理化试验室 Physicochemical Lab
- 仪器分析室 Instrumental Analysis Lab
- 环境试验室 Environmental Lab
- 综合办公室 General Office
- 业务室 Services Office

人员构成



在职员工：48人，研究生学历：6人，大学学历：36人，研究员级高工：5人；高级工程师；6人；工程师：17人。**46 employees of all in the center, 6 persons with master degree, 36 persons with bachelor degree, 5 senior researchers, 6 senior engineers, 17 engineers.**

国家涂料质量监督检验中心
National Quality Supervision Testing Center for Paint

中心主要任务
Primary Duties

- 承担政府指令性产品质量检验任务
  Product quality inspection
- 委托检验 Subcontracted testing
- 国家监督抽查任务
  National supervision and inspection tasks
- 司法鉴定及产品质量仲裁检验
  Judicial identification and product quality arbitration inspection
- 渔业船舶产品认证检验
  Certification and inspection of fishery shipping products
- 3C认证
  China Compulsory Certification
- 环境标志产品认证
  Product Certification of China Environmental Labelling

国家涂料质量监督检验中心
National Quality Supervision Testing Center for Paint

恒温恒湿室
Laboratory of constant temperature-humidity

拉力试验机
Tension tester

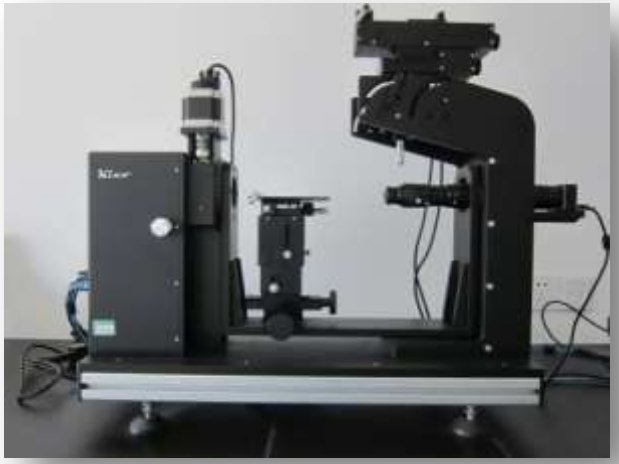
太阳光反射比测试仪
Solar light reflection ratio tester

半球发射率仪
Hemispherical emissivity

全自动抗折抗压试验机
Automatic bending and compression tester

电泳仪
Electrophresis apparatus

国家涂料质量监督检验中心
National Quality Supervision Testing Center for Paint



接触角/表面张力仪
Contact angle/surface tension instrument

高低温湿热试验箱
High and low temperature-humidity test chamber

全自动电位滴定仪
Automatic Potentiometric Titrator

石击试验机
Stone test instrument

导热系数仪
Instrument for Thermal Conductivity

海水浸泡试验箱
Seawater immersion test chamber

国家涂料质量监督检验中心
National Quality Supervision Testing Center for Paint

涂料及原材料有害物质含量分析
Analysis of the content of hazardous substances in coatings and raw materials

涂料及原材料组成的定性、定量分析
Qualitative and quantitative analysis of the composition of coating and raw materials

涂料及原材料结构剖析鉴定
Analysis and identification of coating materials and structure of raw materials

聚合物分子量和分子量分布
Molecular weight and distribution of Polymer

材料热转变温度、热焓和热失重
Thermal transition temperature, enthalpy and thermal weight loss of Materials

材料粒径及粒径分布
Particle size and distribution of materials

材料的微观形貌及元素组成
Micro morphology and elemental composition of the materials

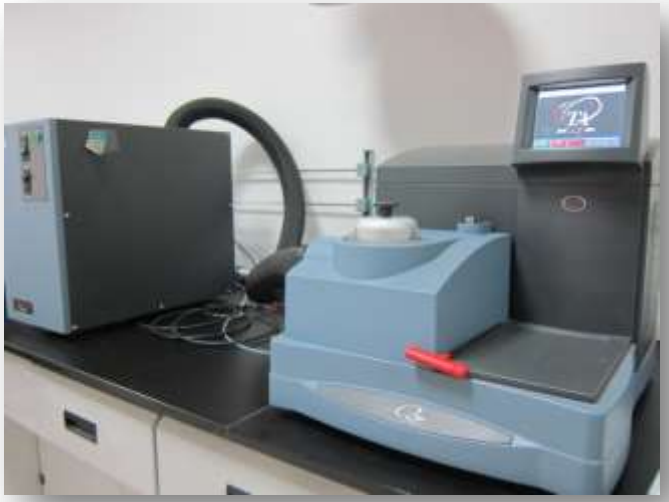
甲醛、VOC测试
Measurement of Methanal and VOCs

气相色谱室
Gas Chromatography laboratory

国家涂料质量监督检验中心
National Quality Supervision Testing Center for Paint

裂解气质联用仪
pyrolysis-GC/MS

扫描电镜
Scanning electron microscopy



差示扫描量热仪
Differential scanning calorimeters

电感耦合等离子发射光谱仪
ICP-OES

国家涂料质量监督检验中心
National Quality Supervision Testing Center for Paint

傅里叶变换显微红外光谱仪
Fourier transform microscopic infrared spectrometer

液相色谱—串联四级杆质谱仪
HPLC/NCI

X射线衍射光谱仪
XRD

数字投射偏光显微镜
Digital projection polarizing microscope

国家涂料质量监督检验中心
National Quality Supervision Testing Center for Paint

- 人工气候老化
  Artificial weathering test
- 耐湿热试验
  Damp and hot resistance test
- 耐中性盐雾试验
  Neutral salt spraying test
- 循环交变腐蚀试验
  Cyclic alternating corrosion test
- 丝状腐蚀
  Filiform corrosion
- 耐冷凝水试验
  Condensation water resistance test
- 模拟压载舱试验Load Simulation test
- 冷凝舱试验
  Condensate simulation test
- 锐边腐蚀
  Sharp edge corrosion

氙灯老化试验机
Xenon tester

加速老化试验箱
Accelerated aging test chamber

湿热试验箱
Thermal-humidity test chamber

盐雾试验箱
Salt-fog tester

冷凝舱模拟系统
Condensate simulation system

压载舱模拟系统
Load Simulation system

感谢各位聆听

Thanks for Listening