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目 次

深圳大气颗粒物中卤代多环芳烃污染研究 ····································	
	3)
北方市曲刑祭放入山, VOC. 排放柱江西京 出版 紀桂貝 何下達 化拉芒 耳石 公太鄉 迷走(152)	2)
北京中央望食区企业 VOUS 非双行证训允	3)
2006~2010年珠三角地区 SO ₂ 特征分析	0)
环境空气 PM、连续监测系统手工采样比对测试 ·························· 王强、钟琪、迟颖、张杨、杨凯(1538	8)
一次 一次 154	4)
然外电产的现在分类型的17000元子,从2015年2月15日中2月中2月中2月中2日中2月中2日中2月中2日中2月中2日中2月中2日中2月中2日中2月中2日中2月中2日中2月中2日中2月中2日中2日中2日中2日中2日中2日中2日中2日中2日中2日中2日中2日中2日中	T)
南海水域不同深及非尤合似生物的直恢浴能及具对不同电于供体的响应	
	0)
基于 GOCI 影像和水体光学分类的内陆湖泊叶绿素 a 浓度遥感估算 冯驰,金琦,王艳楠,赵丽娜,吕恒,李云梅(155)贵州清水江流域丰水期水化学特征及离子来源分析 吕婕梅,安艳玲,吴起鑫,罗进,蒋浩(156)东莞石马河流域水化学特征时空差异及来源辨析 高磊,陈建耀,王江,柯志庭,朱爱萍,许凯(157)	7)
告州清水汀流域主水期水化学蛙征及离子平源分析	5)
贝川伯小红加坡干小河小化于竹皿及两了不断刀切 口灰鸭,又花丝,大尺氅,少处,竹刀(150)	2)
乐完石马河流域水化字特征时至差异及米源辨析 尚磊,陈建雄,土江,柯志庭,朱发泙,计凯(157.	3)
河南鸡冠洞洞穴水对极端气候的响应及其控制因素研究 ·····	
	2.)
石漠化治理对岩溶地下水水化学和溶解无机碳稳定同位素的影响 肖时珍,熊康宁,蓝家程,张晖,杨龙(1590年) 是五元日人以初州西州西州西州西州西州西州西州西州西州西州西州西州西州西州西州西州西州西州西	0)
旱季不同土地利用类型下岩溶碳汇效应差异 赵瑞一,梁作兵,王尊波,于正良,江泽利 (1598	8)
有机氯农药在岩溶区上覆土壤中的垂直迁移特征及对地下水的影响 孙玉川,王永啟,梁作兵,袁道先(160:	5)
山东南四湖沉积物中汞的污染现状及迁移研究 曹霏霏,杨丽原,庞绪贵,王炳华,王云倩(161:	5)
摇蚊幼虫扰动下沉积物微环境和微界面对物理扰动强度的响应 史晓丹,李勇,李大鹏,王忍,邓猛,黄勇(1622	2)
面域列式机列于UMY的风外壳种风介画内切迹机到速反时响应	2)
南 力红壤区 <u></u>	0)
不同紫色母岩对景观水体氮磷及有机物去除的影响 黄雪娇,刘晓晨,李振轮,石纹豪,杨珊(1639	9)
荔枝落叶对铜绿微囊藻牛长和光合作用的影响	8)
带连相节温相物对是菜的丰丽作用	5)
與比似全位使物內球條的母连目中用	5)
南方红壤区氮湿沉降特征及其对流域氮输出的影响	2)
水中利谷隆氯化降解动力学和消毒副产物生成特性 凌晓, 胡晨燕,程明,谷建(1668	8)
化学消毒的中和剂对水中内毒素活性检测的影响 ····································	4)
12 11 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	0)
十美生物灰外小中氨氮的吸附付性	0)
丁二酸改性茶油树木屑吸附铀的研究 张晓峰,陈迪云,彭燕,刘永胜,熊雪莹 (1686	6)
SPG 膜表面润湿性对膜污染和化学耐受性的影响 张静,肖太民,张晶,曹丽亚,杜亚威,刘春,张磊 (1694	4)
TiO 诱导下左旋氨氧沙星的可见光降解及其机制	o)
TiO_2 诱导下左旋氧氟沙星的可见光降解及其机制 郭宏生,刘亚楠,乔琪,魏红,董呈幸,薛洁,李克斌(1700新型高分子絮凝剂对废水中 $Cr(VI)$ 的捕集性能 王刚,杜凤龄,常青,徐敏(1700	7)
利望前分丁系疑剂利及小中Cr(N) 的佣果性能	17)
基于 OUR-HPR 测量在线估计活性污泥合成 PHA 量 曾善文,王泽宇,高敬,刘东,张代钧,卢培利(1713	3)
分离高浓度污泥产酸发酵液的自生动态膜形成机制 ····································	0)
通风强度对市政污泥生物干化中试效果的影响	/
	7)
中脚可吸短数人刘公复歌 N N 一 Z 歌叫bb对没用由重人昆萃取故或的孤家	7)
生物可降解螯合剂谷氨酸 N,N-二乙酸四钠对污泥中重金属萃取效率的研究	
	3)
	3)
	3)
	3)
百乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析············ 田美,刘汉湖,申欣,赵方庆,陈帅,姚永佳(173) 异养硝化-好氧反硝化菌 YL 的脱氮特性····································	3)
百乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析············ 田美,刘汉湖,申欣,赵方庆,陈帅,姚永佳(173) 异养硝化-好氧反硝化菌 YL 的脱氮特性····································	3)
百乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析············ 田美,刘汉湖,申欣,赵方庆,陈帅,姚永佳(173) 异养硝化-好氧反硝化菌 YL 的脱氮特性····································	3)
三百乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析····································	(3) (9) (9) (7) (3) (9)
三百乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析····································	(3) (9) (9) (7) (3) (9)
三年克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析····································	(3) (9) (9) (7) (3) (9)
「具有 上海 一方 上海 上海 上海 上海 上海 上海 上海 上	(3) (9) (7) (3) (9) (6) (5)
三、京、京、京、京、京、京、京、京、京、京、京、京、京、京、京、京、京、京、京	(3) (9) (7) (3) (9) (6) (5)
三乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析	(3) (9) (9) (7) (3) (6) (6) (5) (3)
三乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析	(3) (9) (9) (7) (3) (6) (6) (5) (3)
三乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析	(3) (9) (9) (7) (3) (6) (6) (5) (3)
三乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析	(3) (9) (9) (7) (3) (6) (5) (3)
三乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析	(3) (9) (9) (7) (3) (6) (5) (3)
三乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析	(3) (9) (9) (7) (3) (6) (5) (3)
「日の「日の「日の「日の「日の「日の「日の「日の「日の「日の「日の「日の「日の「	3) 99) 7) 3) 99) 6) 55) 33) 2) 0) 8) 7) 66)
三成街道坐中重金属的分布特征及其健康风险评估 宝鸡市街道坐中逐金属的分布特征及其健康风险评估 宝鸡市街道坐中逐金属的分布转征及其健康风险评估 宝鸡市街道坐埃磁学特征空间分布及环境意义 长期施肥下浙江稻田不同颗粒组分有机碳的稳定特征 宝鸡市街道生块磁学特征空间分布及环境意义 长期施肥下浙江稻田不同颗粒组分有机碳的稳定特征 宝鸡市街道生中重型地类型土壤微生物和酶活性的影响 三江平原典型湿地类型土壤微生物特征与土壤养分的研究 以及,对是,从是有,从是有,从是有,从是有,从是有,从是有,从是有,从是有,从是有,从是	3) 9) 9) 7) 3) 9) 6) 5) 3) 2) 0) 8) 7) 6) 2)
三成街道坐中重金属的分布特征及其健康风险评估 宝鸡市街道坐中逐金属的分布特征及其健康风险评估 宝鸡市街道坐中逐金属的分布转征及其健康风险评估 宝鸡市街道坐埃磁学特征空间分布及环境意义 长期施肥下浙江稻田不同颗粒组分有机碳的稳定特征 宝鸡市街道生块磁学特征空间分布及环境意义 长期施肥下浙江稻田不同颗粒组分有机碳的稳定特征 宝鸡市街道生中重型地类型土壤微生物和酶活性的影响 三江平原典型湿地类型土壤微生物特征与土壤养分的研究 以及,对是,从是有,从是有,从是有,从是有,从是有,从是有,从是有,从是有,从是有,从是	3) 9) 9) 7) 3) 9) 6) 5) 3) 2) 0) 8) 7) 6) 2)
三 三 三 三 三 三 三 三 三 三 三 三 三 三 三 三 三 三 三	3) 9) 9) 7) 3) 9) 6) 5) 3) 2) 0) 8) 7) 6) 2) 6)
「日	3) 99) 7) 33) 99) 66) 55) 33) 2) 00) 88) 77) 66) 22) 99) 66) 44)
「日のLAK 活性汚泥宏基因组的生物多样性及功能分析 田美、刘汉湖、申康、赵方庆、陈帅、姚永佳 (173) 异养硝化-好氧反硝化菌 YL 的脱氮特性 一菜 所	3) 99) 7) 33) 99) 66) 55) 33) 2) (0) 88) 77) 66) 22) 99) 66) 44)
「日のLAK 活性汚泥宏基因组的生物多样性及功能分析 田美、刘汉湖、申康、赵方庆、陈帅、姚永佳 (173) 异养硝化-好氧反硝化菌 YL 的脱氮特性 一菜 所	3) 99) 7) 33) 99) 66) 55) 33) 2) (0) 88) 77) 66) 22) 99) 66) 44)
「日のLAK 活性汚泥宏基因组的生物多样性及功能分析 田美、刘汉湖、申康、赵方庆、陈帅、姚永佳 (173) 异养硝化-好氧反硝化菌 YL 的脱氮特性 一菜 所	3) 99) 7) 33) 99) 66) 55) 33) 2) (0) 88) 77) 66) 22) 99) 66) 44)
「日のLAK 活性汚泥宏基因组的生物多样性及功能分析 田美、刘汉湖、申康、赵方庆、陈帅、姚永佳 (173) 异养硝化-好氧反硝化菌 YL 的脱氮特性 一菜 所	3) 99) 7) 33) 99) 66) 55) 33) 2) (0) 88) 77) 66) 22) 99) 66) 44)
早青、崔延瑞、汤晓晓、杨慧娟、孙剑辉(173: 百乐克(BIOLAK)活性污泥宏基因组的生物多样性及功能分析 田美、刘汉湖、申欣、赵方庆、陈帅、娘永佳(1739	3) 99) 7) 33) 99) 66) 55) 33) 2) (0) 88) 77) 66) 22) 99) 66) 44)
	3) 99) 7) 33) 99) 66) 55) 33) 2) (0) 88) 77) 66) 22) 99) 66) 44)

环境空气 PM25 连续监测系统手工采样比对测试

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摘要: 借鉴美国 EPA 关于 $PM_{2.5}$ 连续监测仪器手工采样比对测试的性能指标和检测方法,结合我国现行的相关技术规范,选取两种不同原理 4 种型号典型的进口和国产环境空气 $PM_{2.5}$ 连续自动监测系统,分别在春、夏、秋、冬这 4 个季节开展了 $PM_{2.5}$ 连续自动监测与手工采样测量(重量法)之间的比对测试. 研究确定了符合我国环境质量现状和环境监测管理需求的 $PM_{2.5}$ 连续监测系统与手工采样比对测试(调试)的质控措施要求(手工采样测量结果平行性 $\le 5 \mu g \cdot m^{-3}$ 或 5%)和技术指标要求(线性回归方程斜率 1 ± 0. 15; 截距 0 ± 10; 相关系数 ≥ 0.95)以及相应的操作方法,为我国环境空气 $PM_{2.5}$ 连续自动监测系统的现场有效使用和数据质量保证提供了必要的技术手段和质控方法.

关键词:PM, 5; 连续监测; 手工采样; 比对测试; 质量控制

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Comparison Test Between PM_{2.5} Continuous Monitoring System and Manual Sampling Analysis for PM_{2.5} in Ambient Air

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Abstract: According to the U. S. EPA performance index and detection methods for comparison test of $PM_{2.5}$ continuous monitoring system, in combination of the current related technical specifications in China, 4 types of typical imported and domestic $PM_{2.5}$ continuous monitoring system with two different principles were tested and compared to the manual sampling measurement (gravimetric method) in spring, summer, autumn and winter. This research determined the quality control requirements (parallelism of manual sampling measurement results $\leq 5 \ \mu g \cdot m^{-3} \ or 5\%$), the technical index (slope of linearity regression equation 1 ± 0.15 ; intercept 0 ± 10 ; correlation coefficient ≥ 0.95), and corresponding detection methods of reference method comparison test to $PM_{2.5}$ continuous monitoring system, which meets the requirement of current environmental quality status and environmental monitoring and management in China. It also provided technical means and method of quality control for effective use and data quality of $PM_{2.5}$ continuous monitoring system in China.

Key words: PM25; continuous monitoring; manual sampling; comparison test; quality control

近年来,我国灰霾天气出现的频次明显增多并 呈区域性特征[1~3]. 2013年1月以来,我国京津冀、 长三角、珠三角等地区多次发生持续的大范围雾 霾,细颗粒物 PM25是导致复合型大气污染问题的核 心污染物[4,5]. PM,5除了造成人体感官上的影响, 更重要的是其对大气有害污染物有明显的富集作 用,尤其是其吸附的有机碳、水溶性离子、重金属 元素等化学成分以及各种病毒细菌等对人体健康及 生态环境形成了极大的威胁[6~10]. 因此,降低大气 PM,,的浓度,改善环境空气质量已经成为各级环境 保护部门的主要任务,而实现对 PM,5的准确监测和 及时预警预报则是当前各级环境监测部门必须完成 的首要工作. 环境空气 PM,,连续自动监测系统是 连续自动监测环境空气中 PM25细颗粒物的实时在 线监测仪器,是对 PM_{2.5}进行准确测量、数据发布、 质量评价以及了解掌握 PM2.5 变化趋势和预警预报 的重要工具. 因此,PM_{2.5}连续自动监测系统的性能质量、功能特点和长期使用的稳定性决定了其能否满足目前环境监测和管理工作的需求,同时也决定了其测量发布的 PM_{2.5}监测数据能否真实反映环境质量现状,监测结果能否与公众的实际感受相一致.由于 PM_{2.5}不像 SO₂等气态污染物容易获取标准物质进行相关溯源校准^[11],因此,环境空气 PM_{2.5}连续自动监测系统安装使用后一般都需要定期与 PM_{2.5}手工采样测量结果进行比对测试或调试,找到连续自动监测与手工采样测量之间的相关关系;通过比对测试或调试对连续自动监测结果进行有效评价或校准修正,这样才能在一定时间段内确保 PM_{2.5}连续

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自动监测系统的监测数据质量,保证自动监测数据 科学可靠,才能进一步使用自动监测数据进行环境 空气质量的评价和相关来源解析等工作^[12-16].

1 美国现行 $PM_{2.5}$ 连续自动监测系统手工采样比对测试要求

美国、欧盟、日本以及国际标准化组织等均

发布过有关环境空气颗粒物(包括 PM_{10} 和 $PM_{2.5}$)自动监测仪器和方法的相关标准和规范 $[17^{-19}]$. 在文献 [17] 中规定了环境空气 $PM_{2.5}$ 连续自动监测系统与手工采样参比测试比对的技术要求,见表 1. 标准中不仅规定了比对测试的技术要求,而且提出相应的检测方法和质控措施要求.

表 1 EPA PART 53 标准中环境空气 PM_{2.5}连续监测系统与手工采样测量比对测试的技术要求

Table 1 Technical indicators for comparing the EPA standard PART 53 recommended PM_{2.5} continuous

monitoring system with manual sampling measurement

项目		性能指标要求		
比对测试的浓厚	3 ~ 200 μg·m ⁻³			
最小检测场均	4			
每个检测场地连续自动监	3			
每个检测场地手工参比多	采样器最小数量	3		
每个检测场地每个季节最小比对	付测试样品数量(天数)	23		
手工参比采样器每组(天)	测试结果的平行性	2 μg·m ⁻³ (SD) 或 5% (RSD)		
连续自动监测仪器每批次测	则试结果的平行性	15% (本批次测试全部样品结果的均方根)		
	相关曲线斜率	1 ± 0. 10		
比对测试手工参比和自动监测结果线 性相关拟合回归曲线	相关曲线截距	0 ±2(最宽要求)		
1.44人18月日归四汉	相关曲线相关系数	≥0.93(最宽要求)		

2 材料与方法

2.1 环境空气 PM_{2.5}连续自动监测系统基本情况 本次比对测试选取了两种不同原理的 4 种型号

进口和国产环境空气 PM25连续自动监测系统,每种

型号自动监测仪器设备各3套,仪器设备基本情况见表2.

2.2 手工采样测量仪器设备

本次比对测试使用的手工采样测量仪器设备情况见表 3.

表 2 测试环境空气 PM_{2.5}连续自动监测系统基本情况

Table 2 Basic information of instrument used in PM _{2.5} continuous monitoring system in ambient air

			2. 3	0 1	
序号	仪器编号	分析原理	进气流量/L·min -1	切割器类型	类型
1	A	β 射线吸收法	16. 67	旋风式	国产
2	В	β 射线吸收法	16. 67	旋风式	国产
3	С	β 射线吸收法	16. 67	旋风式	进口
4	D	振荡天平法	16. 67	旋风式	进口

表 3 测试使用的手工采样测量仪器设备基本情况

Table 3 Basic information of instrument used in PM2 5 manual sampling in ambient air

序号	仪器设备名称	产地	规格、型号
1	小流量 PM _{2.5} 自动换膜采样器(18 台)	美国	2025i(采样流量 16. 67 L·min ⁻¹)
2	流量校准仪	美国	Bios 510
3	温湿度表(校准)	芬兰	HM34
4	空盒气压表	中国	DYM4-1
5	恒温恒湿滤膜自动识别称重系统	德国	AWS-1 电子天平(检定分度值 0.001 mg) 温度控制(20±1)℃ 湿度控制(50±5)% RH

2.3 比对测试实验方法和质控要求

同型号 $PM_{2.5}$ 连续自动监测系统 3 台(套),4 个型号共 12 台(套),同型号手工参比方法 $^{[20,21]}PM_{2.5}$

采样器 18 台,自动监测与参比方法测试同步进行, 采样器与自动监测仪安放位置相距 1 m 左右,采样 入口位于同一高度.取相同采样时间段内的 3 台同 型号自动监测数据的均值($\overline{C_{i,j}}$, $\mu g \cdot m^{-3}$)和18 台采样器手工参比方法采样测试数据的均值($\overline{R_j}$, $\mu g \cdot m^{-3}$)作为一个数据对,i 是仪器的型号序号(i 为 1 ~ 4),j 是有效样品的个数(j 为 1 ~ n, $n \ge 23$),每个样品的采样时间为 23 h,共测试至少 23 个样品.按照以下 5 个步骤进行数据比较.

1540

- (1)每天各台手工采样器同时采样测试的数据结果为一组,计算每组 18 台采样器 $PM_{2.5}$ 手工采样测试样品浓度的平均值 $\overline{R_i}$.
- (2)分别计算每组各台 PM_{2.5} 手工采样器参比 方法测试结果的平行性(标准偏差或相对标准偏 差).
- (3)每天同型号各台自动监测仪与手工参比试相同时间段内的数据结果也为一组,计算每组 PM_{2} ,自动监测仪测试对应时间段内 PM_{3} 。浓度的平均值 $\overline{C_{1}}$.
 - (4)将每组对应的参比测试数据均值分别与各

型号自动监测数据均值组成一组数据对. 每批次比对实验每个型号自动监测仪器至少取得 23 组有效数据对.

(5)将手工参比测试数据与各型号仪器相应的自动监测数据进行线性拟合回归分析,以连续自动监测仪数据为横轴,参比测试数据为纵轴,分别计算4种自动监测仪器本批次比对实验各型号仪器取得回归曲线的斜率(k)、截距(b)和相关系数(r).

3 结果与讨论

3.1 环境空气 PM_{2.5}手工采样测量结果的平行性分析

4 个季节 $PM_{2.5}$ 比对测试 18 台 $PM_{2.5}$ 手工参比 采样仪器手工采样测量共取得数据 119 组. 各季节 手工采样测试样品数量、浓度范围、日均值 ≤ 80 $\mu g \cdot m^{-3}$ 和 > 80 $\mu g \cdot m^{-3}$ 样品的平行性以及当季温湿度情况见表 4.

表 4 4 个季节环境空气 PM_{2.5} 手工采样测量结果的平行性分析

Table 4	Parallelism analysis of	PM _{2.5} data	a by manual	l sampling measuremen	t in four seasons

季节	样品组数	浓度范围		采样器采样 均值)≤80			18 台采样器采样测量结果平均值 (日均值) >80 µg·m ⁻³ 的数据				平均温度	平均相对
学卫	件前组数	$/\mu g \cdot m^{-3}$	组数	各结果的标准偏差/µg·m ⁻³		推偏差/μg·m ⁻³ 组数 各结果的相对标准偏差		圭偏差/%	范围/℃	湿度/%		
			组奴	最大值	最小值	平均值	组奴	最大值	最小值	平均值		
春	28	13. 0 ~ 205. 8	14	4. 9	1.8	3. 5	14	4. 3	1. 9	3. 1	6. 2 ~ 17. 1	52. 5
夏	32	20. 1 ~ 217. 1	22	4. 3	1.4	2. 5	10	3. 5	1.8	2.8	23.0 ~ 31.4	80
秋	28	17. 3 ~ 303. 2	17	3.7	1.3	2. 4	11	4. 1	1. 1	2. 5	8. 1 ~ 18. 7	60. 5
冬	31	22. 0 ~478. 2	6	4.7	1.0	2.6	25	3.6	1.0	2. 1	$-0.2 \sim -8.1$	40

PM_{2.5}手工参比采样测量结果是 PM_{2.5}连续自动监测系统比对测试和调试的基准,因此其多台采样器采样测量结果的平行性是 PM_{2.5}手工比对测试和调试中最重要的质控指标要求.结合表 1 标准中的相关质控要求,分析手工参比采样测量数据的平行性结果如下.

①数据日均值 $\leq 80 \, \mu g \cdot m^{-3}$ 的样品共 59 组,各季节每天手工采样测量结果标准偏差的平均值比较见图 1; 从中看出,在各季节较低浓度($\leq 80 \, \mu g \cdot m^{-3}$)时,手工采样测量结果的平行性(标准偏差)的平均值全部超过表 1 中的要求($\leq 2 \, \mu g \cdot m^{-3}$),但全部结果均小于 5 $\mu g \cdot m^{-3}$.

②数据日均值 > 80 μg·m⁻³的样品共 60 组,各季节每天手工采样测量结果相对标准偏差的平均值比较见图 2;从中看出,在各季节较高浓度(> 80 μg·m⁻³)时,手工采样测量结果的平行性(相对标准偏差)的平均值全部符合表 1 中标准的要求(≤ 5%),且呈现出随着平均浓度逐步升高而手工采样平行性更好的趋势.

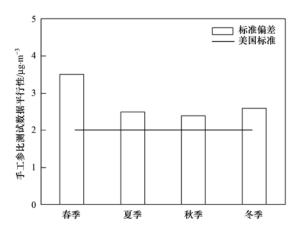


图 1 4 个季节 PM_{2.5} 手工参比采样器采样测量较低浓度 (≤80 µg·m⁻³)数据的平行性

Fig. 1 Parallelism of PM_{2.5} data of lower concentration (\leq 80 μ g·m⁻³) by manual sampling measurement in four seasons

3.2 环境空气 PM_{2.5}连续自动监测系统手工采样比对测试结果分析

将同时间段手工采样分析测量数据和对应的 4 种型号连续自动监测系统测量数据进行线性回归.

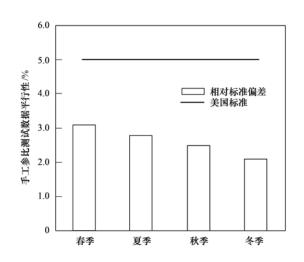


图 2 4 个季节 PM_{2.5} 手工参比采样器采样测量较高浓度 (>80 µg·m⁻³)数据的平行性

Fig. 2 Parallelism of $PM_{2.5}$ data of higher concentration ($>\!80~\mu g\!\cdot\! m^{-3}$) by manual sampling measurement in four seasons

得到每个季节 $PM_{2.5}$ 比对测试的 4 条线性回归曲线; 4 个季节的全部各条回归曲线的斜率 k、截距 b 和相关系数 r 的数值见表 5.

将 4 个季节 4 种型号 PM, 5 连续自动监测系统

的自动监测数据分别与手工参比采样测量数据进行线性回归,所取得回归方程斜率 k 值、截距 b 值以及相关系数 r 的最小值进行比较,结果如图 $3 \sim 5$ 所示. 通过比较分析,可以得到环境空气 $PM_{2.5}$ 连续自动监测系统"手工采样比对测试"或"比对调试"的技术指标要求.

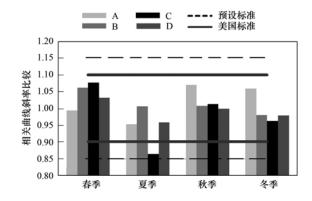


图 3 4 个季节 PM_{2.5} 比对测试手工参比与自动 监测数据线性回归方程斜率比较

Fig. 3 Slope of linearity regression equation of the comparison test between PM_{2.5} manual sampling measurement and PM_{2.5} continuous monitoring system in four seasons

表 5 4 种型号 $PM_{2.5}$ 连续自动监测系统 4 个季节手工采样比对测试结果分析

Table 5 Analysis of the comparative test results of PM_{2.5} manual sampling measurement and 4 types of PM_{2.5} continuous monitoring systems in four seasons

				1 1112.5 COL	muous mon	morning by bi	cino in rou	БСизопо				
回归曲线		春季			夏季			秋季			冬季	
四归四线	k	b	r	k	b	r	k	b	r	k	b	r
A	0. 994	4. 171	0. 991	0. 953	- 6. 597	0. 990	1.070	- 8. 145	0. 996	1. 059	2. 402	0. 996
В	1.062	-3.995	0. 988	1.007	-7.627	0. 982	1.008	-7.038	0. 996	0.980	2. 523	0. 998
С	1. 076	0.480	0.990	0.863	- 0. 627	0. 987	1.014	-6.693	0. 988	0.962	1. 946	0. 987
D	1. 031	-2.018	0. 990	0. 959	-4. 229	0. 993	0. 998	-5.450	0. 997	0. 979	3. 688	0. 998

结合表 4 中的环境温湿度条件,从图 3~5 中可以看出:①针对斜率 k,大部分值符合表 1 中(1±0.1)的技术指标要求;其中夏季不同型号仪器间波动较大.最小值小于 0.9,分析原因可能是夏季与春、秋和冬季相比,PM_{2.5}整体浓度相对较低,且夏季比对时环境温度相对较高,湿度相对较大;在高温高湿环境下,存在颗粒的损失(如高温挥发)或吸湿(如盐类潮解)等导致手工采样和自动测量的颗粒浓度变化的情况.使得仪器比对测量或调试时系统误差比较明显;②针对截距 b,只有 3 个数据符合表 1 中(0±2)的技术指标要求,其余各季节全部超出;其中夏季和秋季的最大偏差值接近 8.分析原因可能还是由于我国目前 PM_{2.5}整体基础浓度较高,浓度波动较大,组成成分复杂,造成比对测量本底随机误差较大.③针对相关系数 r,全部数据均符合表

1 中(>0.93)的技术指标要求,且全部大于 0.95. 尤其是秋季和冬季,基本能够达到 0.99,分析原因可能是因为在秋、冬季,由于燃煤取暖我国环境空气 PM_{2.5}浓度普遍较高、跨度较大,且环境相对湿度较低,有利于仪器线性相关拟合,消除或减小误差影响,提高比对测量的相关性.

4 结论

(1)环境空气 $PM_{2.5}$ 连续监测系统比对测试(调试)中,"手工参比采样测量结果的平行性"指标是其中首要的质控指标要求,是 $PM_{2.5}$ 比对测试数据有效的前提. 4 个季节的比对测试结果发现由于我国目前环境空气 $PM_{2.5}$ 的基础浓度较国外高,浓度波动较大,且各季节温湿度变化较大, $PM_{2.5}$ 成分也有差异;因此当浓度较低时($\leq 80 \mu g \cdot m^{-3}$)可适当放宽

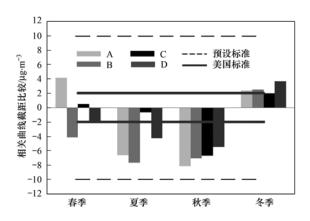


图 4 4 个季节 PM_{2.5} 比对测试手工参比与自动 监测数据线性回归方程截距比较

Fig. 4 Intercept of linearity regression equation of the comparison test between $PM_{2.5}$ manual sampling measurement and $PM_{2.5}$ continuous monitoring system in four seasons

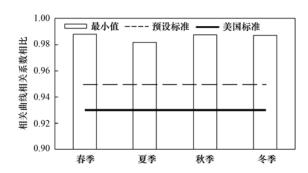


图 5 4 个季节 PM_{2.5} 比对测试手工参比与自动 监测数据线性回归方程相关系数比较

Fig. 5 Correlation coefficient of linearity regression equation of the comparison test between PM_{2.5} manual sampling measurement and PM_{2.5} continuous monitoring system in four seasons

手工采样测量的平行性要求. $PM_{2.5}$ 手工比对测试 (调试)质控指标要求为"至少3台以上采样器每组 参比采样测量结果的标准偏差 $\leq 5 \mu g \cdot m^{-3}$ 或相对标准偏差 $\leq 5\%$ ".

(2)受不同地域分布和气候特点的影响,现场安装使用的环境空气 $PM_{2.5}$ 连续监测系统必须与手工采样测量进行有效的比对测试或调试,符合相关校准技术要求后,才能确保其 $PM_{2.5}$ 连续自动监测数据准确可靠,真实反映当地的 $PM_{2.5}$ 污染状况. 在现有 $PM_{2.5}$ 污染状况等实际条件下,不宜直接照搬国外标准,目前" $PM_{2.5}$ 手工比对测试(调试)"技术指标要求可初步设置为: $PM_{2.5}$ 连续自动监测系统与手工采样测量比对,取得相关校准曲线: 斜率 k 为(1 ± 0.15),截距 k 为(0 ± 10),相关系数 r 为(\geqslant 0.95);

随着 PM_{2.5}污染的逐步治理,相关要求可结合我国国情逐步加严.

(3)本次 $PM_{2.5}$ 连续自动监测系统手工采样比对测试的检测过程和方法借鉴了美国 EPA PART 53 标准,比对测试研究工作覆盖一年四季典型的不同天气,测试现场 $PM_{2.5}$ 浓度范围较宽(13.0~478.2 $\mu g \cdot m^{-3}$),比对测试结果的代表性较强. 因此,本次比对测试获得的质控和技术指标完全可以同样应用于对环境空气 $PM_{2.5}$ 连续自动监测系统仪器的性能质量评价和日常运行管理,以满足环境监测和管理的技术需求.

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《环境科学》自创刊以来,始终坚持"防治污染,改善生态,促进发展,造福人民"的宗旨,报道我国环境科学领域内具有创新性高水平,有重要意义的基础研究和应用研究成果,以及反映控制污染,清洁生产和生态环境建设等可持续发展的战略思想、理论和实用技术等.

《环境科学》在国内外公开发行,并在国内外科技界有较大影响,被国内外一些重要检索系统收录,如美国医学索引 MEDLINE;美国化学文摘 CA;俄罗斯文摘杂志 AJ;美国生物学文摘预评 BP;美国医学索引 IM;日本科学技术情报中心数据库 JICST;英国动物学记录 ZR;剑桥科学文摘(CSA):Environmental Sciences;剑桥科学文摘(CSA):Pollution Abstracts;剑桥科学文摘(CAS):Life Sciences Abstracts等;国内的检索系统有中国科技论文统计与引文数据库(CSTPCD);中文科技期刊数据库(维普);中国期刊全文数据库(CNKI);数字化期刊全文数据库(万方);中国科学引文数据库(CSCD);中国生物学文摘等.

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CONTENTS

Pollution of Halogenated Polycyclic Aromatic Hydrocarbons in Atmospheric Particulate Matters of Shenzhen	
Emission Characteristics of VOCs from Typical Restaurants in Beijing	
Characteristics Analysis of Sulfur Dioxide in Pearl River Delta from 2006 to 2010	
Comparison Test Between PM _{2.5} Continuous Monitoring System and Manual Sampling Analysis for PM _{2.5} in Ambient Air Determination and Emission of Condensable Particulate Matter from Coal-fired Power Plants	WANG Qiang, ZHONG Qi, CHI Ying, et al. (1538)
Potential Carbon Fixation Capability of Non-photosynthetic Microbial Community at Different Depth of the South China Sea and Its	
rotential Carbon Fixation Capability of Non-photosynthetic successful Community at Different Depth of the South China Sea and its	Response to Different Electron Donois
Remote Sensing Estimation of Chlorophyll-a Concentration in Inland Lakes Based on GOCI Image and Optical Classification of Wat	
Hydrochemical Characteristics and Sources of Qingshuijiang River Basin at Wet Season in Guizhou Province	
Temporal-spatial Variation and Source Identification of Hydro-chemical Characteristics in Shima River Catchment, Dongguan City	
Response and Control Factors of Groundwater to Extreme Weather, Jiguan Cave, Henan Province, China	
Impact of Rocky Desertification Treatment on Underground Water Chemistry and Dissolved Inorganic Carbon Isotope in Karst Areas	······
Difference of Karst Carbon Sink Under Different Land Use and Land Cover Areas in Dry Season Vertical Migration Characteristics of Organochlorine Pesticides in Overlying Soil in Karst Terranes and Its Impact on Groundwater	
	·· SUN Yu-chuan, WANG Yong-qi, LIANG Zuo-bing, et al. (1605)
Pollution Status and Migration of Mercury in the Sediments of Nansi Lake in Shandong Province	
Response of Sediment Micro Environment and Micro Interface to Physical Disturbance Intensity Under the Disturbance of Chironom	nus plumosus ·····
Characteristics of Atmospheric Nitrogram Was Describing and Associated Laurest on N. Tayannest in the Wastenhald of Ded Scil Associated	
Characteristics of Atmospheric Nitrogen Wet Deposition and Associated Impact on N Transport in the Watershed of Red Soil Area i	
Effect of Different Purple Parent Rock on Removal Rates of Nitrogen, Phosphorus and Organics in Landscape Water	
Effects of Litchi chinensis Defoliation on Growth and Photosynthesis of Microcystis aeruginosa	
Effects of Literal crunensis Detoliation on Growth and Photosynthesis of Microcystis aeruginosa Toxicity of Coptis chinensis Rhizome Extracts to Green Algae	WANG Alao-xiong, JIANG Chen-chun, Li Jin-wei, et al. (1048)
Formation Mechanism of the Disinfection By-product 1,1-Dichloroacetone in Drinking Water	
Degradation Kinetics and Formation of Disinfection By-products During Linuron Chlorination in Drinking Water	
Interference for Various Quench Agents of Chemical Disinfectants on Detection of Endotoxin Activities in Water	
Ammonium Adsorption Characteristics in Aqueous Solution by Dairy Manure Biochar	
Absorption of Uranium with Tea Oil Tree Sawdust Modified by Succinic Acid	
Effect of Membrane Wettability on Membrane Fouling and Chemical Durability of SPG Membranes	
TiO ₂ -Induced Photodegradation of Levofloxacin by Visible Light and Its Mechanism	
Performance of Novel Macromolecule Flocculant in the Treatment of Wastewater Containing Cr(VI) Ions	
On-line Estimation for the Amount of Stored PHA in Activated Sludge Based on OUR-HPR Measurements	
Formation Mechanism of Self-forming Dynamic Membrane During Separation of High-concentration Sewage Sludge Fermented for Ac	cia Froduction
	HILANC Shari LIII Hang be VIN Do at al. (1720)
Influence of Air Elius on Municipal Studeo Biodesine in a Dilat Scale Test	HUANG Shuai, LIU Hong-bo, YIN Bo, et al. (1720)
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test	HUANG Shuai, LIU Hong-bo, YIN Bo, et al. (1720) ZHANG Yu, HAN Rong, LU Wen-jing, et al. (1727)
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Bretibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis (carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Breeibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe	HUANG Shuai, LIU Hong-bo, YIN Bo, et al. (1720) ZHANG Yu, HAN Rong, LU Wen-jing, et al. (1727) WU Qing, CUI Yan-rui, TANG Xiao-xiao, et al. (1733) TIAN Mei, LIU Han-hu, SHEN Xin, et al. (1739) LIANG Xian, REN Yong-xiang, YANG Lei, et al. (1749) REN Lei, SHI Yan-hua, JIA Yang, et al. (1757) LIU Zhi-chen, YE Jin-shao, PENG Hui, et al. (1763) LI Lei, XU Jing, ZHAO You-cai, et al. (1769) of Different Configurations MAI Xiao-bei, TAO Ran, YANG Yang, et al. (1776) LIU Rong, LONG Yan, WANG Li-li, et al. (1785) YAN Jun-xia, LI Hong-jian, LI Jun-jian, et al. (1793)
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau	HUANG Shuai, LIU Hong-bo, YIN Bo, et al. (1720) ZHANG Yu, HAN Rong, LU Wen-jing, et al. (1727) WU Qing, CUI Yan-rui, TANG Xiao-xiao, et al. (1733) TIAN Mei, LIU Han-hu, SHEN Xin, et al. (1739) LIANG Xian, REN Yong-xiang, YANG Lei, et al. (1749) REN Lei, SHI Yan-hua, JIA Yang, et al. (1757) LIU Zhi-chen, YE Jin-shao, PENG Hui, et al. (1763) LI Lei, XU Jing, ZHAO You-cai, et al. (1769) of Different Configurations MAI Xiao-bei, TAO Ran, YANG Yang, et al. (1776) LIU Rong, LONG Yan, WANG Li-li, et al. (1785) YAN Jun-xia, LI Hong-jian, LI Jun-jian, et al. (1793)
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N,N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N,N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dusts in Xuanwei and Their Health Risk Assessment	HUANG Shuai, LIU Hong-bo, YIN Bo, et al. (1720) ZHANG Yu, HAN Rong, LU Wen-jing, et al. (1727) WU Qing, CUI Yan-rui, TANG Xiao-xiao, et al. (1733) TIAN Mei, LIU Han-hu, SHEN Xin, et al. (1739) LIANG Xian, REN Yong-xiang, YANG Lei, et al. (1749) REN Lei, SHI Yan-hua, JIA Yang, et al. (1757) LIU Zhi-chen, YE Jin-shao, PENG Hui, et al. (1763) LI Lei, XU Jing, ZHAO You-cai, et al. (1769) of Different Configurations MAI Xiao-bei, TAO Ran, YANG Yang, et al. (1776) LIU Rong, LONG Yan, WANG Li-li, et al. (1785) YAN Jun-xia, LI Hong-jian, LI Jun-jian, et al. (1793) Sess Plateau JIANG Ji-shao, GUO Sheng-li, WANG Rui, et al. (1802) ZHANG Wen-chao, LÜ Sen-lin, LIU Ding-yu, et al. (1810)
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis (carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dust in Baoji City and Its Implications of Environment Effect of Long-term Fertilizer Application on the Stability of Organic Carbon in Particle Size Fractions of a Paddy Soil in Zhejiang I	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N,N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dusts in Xuanwei and Their Health Risk Assessment Spatial Distribution of Magnetic Properties of Street Dust in Baoji City and Its Implications of Environment Effect of Long-term Fertilizer Application on the Stability of Organic Carbon in Particle Size Fractions of a Paddy Soil in Zhejiang I	HUANG Shuai, LIU Hong-bo, YIN Bo, et al. (1720) ZHANG Yu, HAN Rong, LU Wen-jing, et al. (1727) WU Qing, CUI Yan-rui, TANG Xiao-xiao, et al. (1733) TIAN Mei, LIU Han-hu, SHEN Xin, et al. (1739) LIANG Xian, REN Yong-xiang, YANG Lei, et al. (1749) REN Lei, SHI Yan-hua, JIA Yang, et al. (1763) LIU Zhi-chen, YE Jin-shao, PENG Hui, et al. (1763) LIU Lei, XU Jing, ZHAO You-cai, et al. (1769) of Different Configurations MAI Xiao-bei, TAO Ran, YANG Yang, et al. (1776) YAN Jun-xia, LI Hong-jian, LI Jun-jian, et al. (1785) YAN Jun-xia, LI Hong-jian, LI Jun-jian, et al. (1793) SES Plateau ZHANG Ji-shao, GUO Sheng-li, WANG Rui, et al. (1802) ZHANG Wen-chao, LÜ Sen-lin, LIU Ding-yu, et al. (1810) ZHANG Jun-hui, WANG Jin, ZHANG Jian, et al. (1818) Province, China MAO Xia-li, LU Kou-ping, SUN Tao, et al. (1827)
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N,N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dusts in Xuanwei and Their Health Risk Assessment Spatial Distribution of Magnetic Properties of Street Dust in Baoji City and Its Implications of Environment Effect of Long-term Fertilizer Application on the Stability of Organic Carbon in Particle Size Fractions of a Paddy Soil in Zhejiang I	HUANG Shuai, LIU Hong-bo, YIN Bo, et al. (1720) ZHANG Yu, HAN Rong, LU Wen-jing, et al. (1727) WU Qing, CUI Yan-rui, TANG Xiao-xiao, et al. (1733) TIAN Mei, LIU Han-hu, SHEN Xin, et al. (1739) LIANG Xian, REN Yong-xiang, YANG Lei, et al. (1749) REN Lei, SHI Yan-hua, JIA Yang, et al. (1763) LIU Zhi-chen, YE Jin-shao, PENG Hui, et al. (1763) LIU Lei, XU Jing, ZHAO You-cai, et al. (1769) of Different Configurations MAI Xiao-bei, TAO Ran, YANG Yang, et al. (1776) YAN Jun-xia, LI Hong-jian, LI Jun-jian, et al. (1785) YAN Jun-xia, LI Hong-jian, LI Jun-jian, et al. (1793) SES Plateau ZHANG Ji-shao, GUO Sheng-li, WANG Rui, et al. (1802) ZHANG Wen-chao, LÜ Sen-lin, LIU Ding-yu, et al. (1810) ZHANG Jun-hui, WANG Jin, ZHANG Jian, et al. (1818) Province, China MAO Xia-li, LU Kou-ping, SUN Tao, et al. (1827)
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N,N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dusts in Xuanwei and Their Health Risk Assessment Spatial Distribution of Magnetic Properties of Street Dust in Baoji City and Its Implications of Environment Effect of Long-term Fertilizer Application on the Stability of Organic Carbon in Particle Size Fractions of a Paddy Soil in Zhejiang I Effects of Different Reclaimed Scenarios on Soil Microbe and Enzyme Activities in Mining Areas	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dusts in Xuanwei and Their Health Risk Assessment Spatial Distribution of Magnetic Properties of Street Dust in Baoji City and Its Implications of Environment Effects of Different Reclaimed Scenarios on Soil Microbe and Enzyme Activities in Mining Areas Soil Microorganism Characteristics and Soil Nutrients of Different Wetlands in Sanjinag Plain, Northeast China	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N,N-bis(carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dust in Baoji City and Its Implications of Environment Effect of Long-term Fertilizer Application on the Stability of Organic Carbon in Particle Size Fractions of a Paddy Soil in Zhejiang I Effects of Different Reclaimed Scenarios on Soil Microbe and Enzyme Activities in Mining Areas Soil Microorganism Characteristics and Soil Nutrients of Different Wetlands in Sanjinag Plain, Northeast China Strengthening Effects of Sodium Salts on Washing Kerosene Contaminated Soil with Surfactants	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis (carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dusts in Xuanwei and Their Health Risk Assessment Spatial Distribution of Magnetic Properties of Street Dust in Baoji City and Its Implications of Environment Effect of Long-term Fertilizer Application on the Stability of Organic Carbon in Particle Size Fractions of a Paddy Soil in Zhejiang I Effects of Different Reclaimed Scenarios on Soil Microbe and Enzyme Activities in Mining Areas Soil Microorganism Characteristics and Soil Nutrients of Different Wetlands in Sanjinag Plain, Northeast China Strengthening Effects of Sodium Salts on Washing Kerosene Contaminated Soil with Surfactants Effects and Biological Response on Bioremediation of Petroleum Contaminated Soil	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis (carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dusts in Xuanwei and Their Health Risk Assessment Spatial Distribution of Magnetic Properties of Street Dust in Baoji City and Its Implications of Environment Effect of Long-term Fertilizer Application on the Stability of Organic Carbon in Particle Size Fractions of a Paddy Soil in Zhejiang I Effects of Different Reclaimed Scenarios on Soil Microbe and Enzyme Activities in Mining Areas Soil Microorganism Characteristics and Soil Nutrients of Different Wetlands in Sanjinag Plain, Northeast China Strengthening Effects of Sodium Salts on Washing Kerosene Contaminated Soil with Surfactants Effects and Biological Response on Bioremediation of Petroleum Contaminated Soil Enhanced Phytoextraction of Heavy Metals from Contaminated Soils Using Sedum alfredii Hance with Biodegradable Chelate GLDA	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis (carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dust in Baoji City and Its Implications of Environment Effect of Long-term Fertilizer Application on the Stability of Organic Carbon in Particle Size Fractions of a Paddy Soil in Zhejiang I Effects of Different Reclaimed Scenarios on Soil Microbe and Enzyme Activities in Mining Areas Soil Microorganism Characteristics and Soil Nutrients of Different Wetlands in Sanjinag Plain, Northeast China Strengthening Effects of Sodium Salts on Washing Kerosene Contaminated Soil with Surfactants Effects and Biological Response on Bioremediation of Petroleum Contaminated Soil Enhanced Phytoextraction of Heavy Metals from Contaminated Soils Using Sedum alfredii Hance with Biodegradable Chelate GLDA Speciation Characteristics and Bioavailability of Heavy Metals in Oasis Soil Under Pb, Zn Combined Stress	
Influence of Air Flux on Municipal Sludge Biodrying in a Pilot Scale Test Extraction of Heavy Metals from Sludge Using Biodegradable Chelating Agent N, N-bis (carboxymethyl) Glutamic Acid Tetrasodium Biodiversity and Function Analyses of BIOLAK Activated Sludge Metagenome Characteristics of Nitrogen Removal by a Heterotrophic Nitrification-Aerobic Denitrification Bacterium YL Biodegradation Characteristics and Kinetics of p-nitrophenol by Strain Arthrobacter sp. CN2 Biodegradation of Pyrene by Intact Cells and Spores of Brevibacillus brevis Investigation of Antibiotic Resistance Genes (ARGs) in Landfill Investigation of Antibiotic Resistance of Indigenous Bacteria and Abundance of Class I Integron in Matrix of Constructed Wetlands Effects of Nitrate and CH ₄ on Anaerobic Oxidation of BETX in Landfill Cover Soils Spatial Heterogeneity of Soil Respiration in a Planted Larch Forest in Shanxi Plateau Effects of Nitrogen Fertilization on Soil Respiration and Temperature Sensitivity in Spring Maize Field in Semi-Arid Regions on Loe Distribution Characteristics of Heavy Metals in the Street Dusts in Xuanwei and Their Health Risk Assessment Spatial Distribution of Magnetic Properties of Street Dust in Baoji City and Its Implications of Environment Effect of Long-term Fertilizer Application on the Stability of Organic Carbon in Particle Size Fractions of a Paddy Soil in Zhejiang I Effects of Different Reclaimed Scenarios on Soil Microbe and Enzyme Activities in Mining Areas Soil Microorganism Characteristics and Soil Nutrients of Different Wetlands in Sanjinag Plain, Northeast China Strengthening Effects of Sodium Salts on Washing Kerosene Contaminated Soil with Surfactants Effects and Biological Response on Bioremediation of Petroleum Contaminated Soil Enhanced Phytoextraction of Heavy Metals from Contaminated Soils Using Sedum alfredii Hance with Biodegradable Chelate GLDA	

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