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COMMITTEE ON COMMODITY PROBLEMS INTERGOVERNMENTAL GROUP ON TEA Nineteenth Session

New Delhi, India, 12 – 14 May 2010

DEVELOPMENT OF TEA INDUSTRY IN CHINA

中国茶叶代表团

The delegation on Tea of China

参加粮农组织政府间茶叶工作组 第 19 届会议文件

Report for the Nineteenth Session of FAO Intergovernmental Working Group Meeting on Tea

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中国的茶叶产业发展状况

一、总论

中国是世界上发现和利用茶树最早的国家,是举世公认的穿的故乡。从世界范围来看,中国是目前全球最大的茶叶生产国利消费国,茶叶种植面积和产量连续多年位居世界第一,在世界茶产业中居于主导地位。全国有20个省、自治区、直辖市生产茶叶,涉茶人员约8000万人。茶叶是中国重要的经济作物之一,茶产业的发展对于中国产茶区的经济发展、农民致富具有十分重要的作用在下面的分析中将详细介绍近年来中国茶产业发展情况。

二、国内供应、需求和贸易情况

(一)生产:种植面积、收获面积及产量

2009年中国茶叶种植面积达到了186.7万公顷,开采面积为138万公顷,分别比2008年增长6.9%和6.6%;茶叶总产量为13.万吨,增长7%;茶叶总产值413.6亿元,增长16.9%。中国茶叶生产面积、产量和产值总体上是持续、稳定增长,其中2004年以来增长速度更快(图1)。

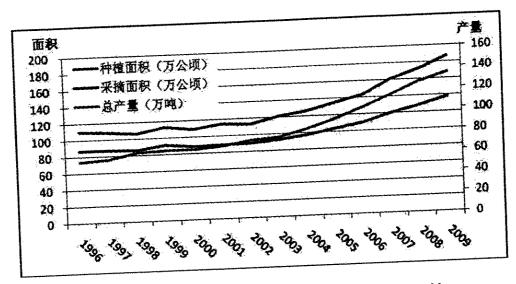


图 1 中国茶叶生产面积与产量变化趋势

从生产的茶类结构看,中国主要以生产绿茶为主。2008年绿茶、乌龙茶、红茶三大茶类产量分别为92.7万吨、14.4万吨和6.97万吨,分别占总产量的74%、11%和6%,可见绿茶居于绝对主导地位。从变化趋势看(图2),中国乌龙茶产量呈现不断增长的趋势,红茶产量从1998年到2003年总体是连续下降,2004年开始产量不断提高,紧压茶的产量近年来比较稳定,其他茶类的总产量随着消费需求的多元化近年来也出现较快的增长势头。

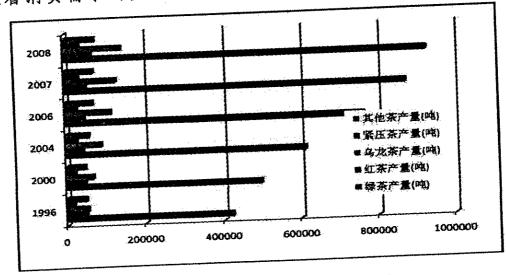


图 2 中国茶叶生产的产品结构

(二)貿易:出口/再出口和进口

近年来中国茶叶出口总量平稳增长、分种类有增有减,绿茶、乌龙茶、白茶出口增加,红茶、黑茶和其他特种茶出口减少(图3)。 2009年茶叶出口30.3万吨(表1),出口金额7.05亿美元。从出口结构看,绿茶出口22.93万吨,金额5.25亿美元;红茶出口4万多吨,金额6437万美元;乌龙茶出口2.4万吨,金额6686万美元;花茶出口5914吨,金额2963万美元。

表 1	近年来中	国茶叶进出	【口情况	单位:万吨	、亿美元
					• 16

年份	出口数量	出口金额	进口数量	进口金额
1999	19.96	3.38	0. 19	0.03
2000	22.77	3.47	0. 24	0.04
2001	24. 97	3.42	0.17	0.03
2002	25.23	3.32	0. 17	0.03
2003	25.99	3.67	0.29	0.04
2004	28.02	4.37	0.23	0.06
2005	28.66	4. 84	0.28	0.07
2006	28.66	5.47	0.32	0.08
2007	28.95	6.08	0.53	0.13
2008	29.69	6.82	0.54	0.18
2009	30.3	7.05		

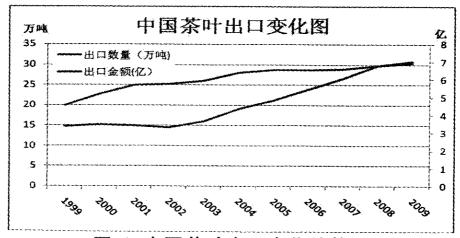


图 3 中国茶叶出口变化趋势图

近年来中国茶叶进口量在不断扩大,同时进口金额也快速增长。2008年中国茶叶贸易总量30.23万吨,其中进口量5398吨。进口以红茶为主,2008年进口红茶4306吨,占茶叶进口量的79.77%;绿茶进口944吨,占17.49%。

(三)消费

中国是茶叶消费大国,近年来随着居民收入水平的显著提高,全国茶叶消费量以每年10%至15%的速度在不断增长,估计2009年中国茶叶总消费量达到100万吨。在中国的主要茶叶消费区域茶叶消费量增加更为明显,一项调查表明中国广州年人均茶叶消费量高达2.50公斤。

三、销售渠道

(一)当前中国茶叶销售渠道的总体介绍

20世纪80年代中期,中国对国内茶叶流通体制进行了改革。 当前中国国内茶叶的基本流通渠道如图4。其中花茶、边销茶经过专门精制后进入市场,而以中高档绿茶为主的名优茶则主要通过个体茶农和茶场加工后直接进入市场。在生产者与消费者之间存在若干类型的中间经济组织,主要包括大众市场网络、茶叶专业经销商和茶叶交易市场。部分茶叶直接从生产者到消费者,部分茶叶需要经过中间经济组织才能到达消费者手中。在中国茶叶交易市场是名优茶交易的主要途径,茶叶交易市场的参与主体一般包括生产者、中间组织及茶庄等。

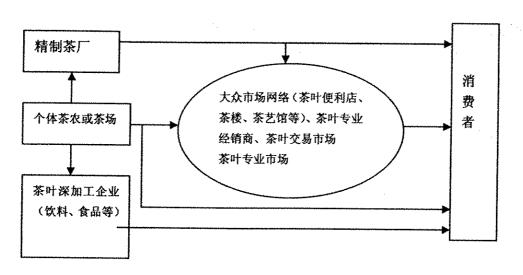


图 4 当前中国内茶叶流通渠道

(二)茶农的销售渠道

根据中国茶叶产业技术体系产业经济研究室在浙江、云南、福建等10个茶叶主产省调查的518户茶农的数据,我们发现当前把茶叶卖给当地的龙头企业是目前最主要的茶叶销售方式,通过该方式的茶农户数占总数的46.3%;排在第二位的销售方式是交给中间商,单纯采用该方式的茶农占20.3%(涉及该方式的占26.1%。);第三种方式是自己在当地农贸市场出售,占11.8%;第四种方式通过专业合作社销售茶叶的占6.4%(涉及合作社的茶农占12.4%)(表2)。由此可以可见,茶农销售茶叶方式已经发生了根本性转变,各级龙头企业发挥了积极的作用,但传统的销售渠道(农贸市场)依然占较大的比例。

表 2 茶农的茶叶销售渠道

	茶农户数	比例
销售方式	61	11.8%
①自己在农贸市场销售	105	20.3%
②交给中间商	240	46.3%
③交给龙头企业	33	6.4%
④通过合作组织 ⑤在自己开设的茶叶店出		2.0%
1 售	15	2.9%
含①的	95	18.3%
含②的	135	26.1%
含⑤的	26	5.0%
含③的	270	52.1%
含④的	64	12.4%
含①②的	215	41.5%

(三)茶叶企业的销售渠道

根据中国茶叶产业技术体系产业经济研究室 2009 年的一项调查,目前中国茶叶企业的营销渠道选择行为有两个突出特点,一是倾向于自建渠道,有74%的受访企业倾向于自建营销渠道和销售网络。二是多渠道并用,统计数据显示65%的企业采取的是多渠道的销售模式(表3)。初步分析,导致这两个特点的原因是经营观念和利益分配问题。目前茶叶零售环节被认为是利润最丰厚的环节,因此为了获得更多的销售利润,更多的企业必然选择自建销售网络或通过其他方式控制销售终端。

表 3 茶叶销售渠道

渠道与渠道组合	企业个数	比例
①自建销售网点	45	22%
②借助商超渠道	1	0%
③委托或合同销售	6	3%
④通过批发市场	4	2%
⑤等客户主动购买	10	5%
两个销售渠道	58	29%
三个以上渠道	74	36%
涉及①的	151	74%
涉及②的	66	33%
涉及③的	83	41%
涉及⑤的	81	40%

四、市场份额分析

(一)批发零售渠道

中国茶叶市场份额主要在国内,占整个茶叶销售量的77.8%,国外市场约占22.2%。国内市场大多经过批发到零售。批发市场分产地和销地。产地批发市场目前比较少,只有茶叶产量比较多的县建有;销地批发市场目前比较多,全国大中城市基本上都有,其中北京、上海、天津、广州、武汉、沈阳等特大型城市,茶叶批发市场规模都比较大。这些批发市场一般也兼零售。中国茶叶零售分销渠道中,茶叶专卖店和产地交易市场是茶叶的主要分销渠道。原因是这些地方选购茶叶消费者可以事先品尝不同价位、口味、色泽的茶叶,选择的空间大,能够满足不同消费者的偏好。特别是在茶产地,茶叶供应数量较多,销售商之间的竞争激烈,市场

信息也比较完全、透明,消费者可以与茶叶销售商进行讨价还价, 最终选购到自己喜欢的类型和价格的茶叶。而在超市或者商场里 面购买茶叶时不可能让消费者事先品尝斟酌或者讲价。在茶馆消 费则侧重休闲体验,优越舒适的环境、高附加价值的服务使得茶叶 价格相对较高。根据调查结果显示,茶叶产区和销区的消费者愿 意选择的渠道模式有所不同。在产区,消费者更愿意选择直接从 茶农那里购买茶叶。在产地购买茶叶,可通过观察茶园的环境和 加工过程,判断茶叶的品质,现场体验消费后再做出购买决策;而 销区的消费者为便利和品质保障起见,更愿意选择在茶叶专卖店 消费。伴随茶叶品牌连锁店的迅速扩张,茶叶越来越向品牌和大 户集中,市场信息和物流的进步带来了产区茶叶批发市场的进一 步繁荣,大的茶叶销售企业直接到产区采购茶叶,有的甚至在产区 建立自己的生产基地,以减少茶叶原料贸易环节,降低生产经营成 本。

(二)适用原产地规则的进口份额

中国茶叶受到世界各国消费者的欢迎,截止到2009年中国茶叶已出口到全球110多个国家和地区,中国茶叶进口量前8位国家占中国茶叶出口总量的60%以上。中国绿茶以摩洛哥、美国、乌兹别克斯坦、日本、巴基斯坦、阿尔及利亚、马里、尼日利亚、塞内加尔、突尼斯、香港等国家和地区为主要市场,红茶主要出口到俄罗斯、美国、波兰、英国、香港和巴基斯坦等国家和地区,特种茶—8—

(以乌龙茶和花茶为主)主销日本。摩洛哥、日本、乌兹别克斯坦、 美国和俄罗斯是中国主要的茶叶进口国。近年来摩洛哥一直是中 国茶叶第一大进口国,2009年从中国进口量达 5.85万吨;乌兹别 克斯坦位居第二,进口量 2.2万吨;俄罗斯上升为中国茶叶出口第 三大市场,进口量达 2.1万吨;美国受金融危机的影响位居第四, 从中国进口量 1.9万吨;日本 2009年从中国进口的茶叶继续呈下 降态势,已跌至不到 1.9万吨;巴基斯坦从中国进口量 1.48万吨 (图 5)。

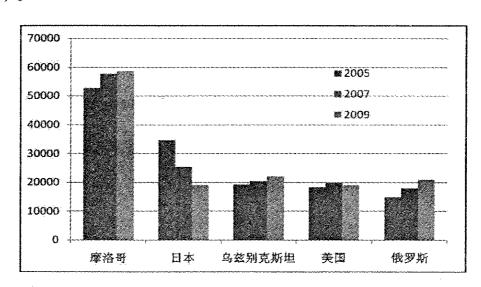


图 5 中国茶叶主要进口国进口量的变化

(三)产品类别

从消费茶类看目前中国茶叶消费以绿茶为主,2006年绿茶消费量为38.5万吨,占国内茶叶总消费量的58%;乌龙茶与花茶消费量已经接近,各为8万吨左右,各占国内茶叶总消费量的12%;红茶消费量为1.5万吨,约占国内茶叶总消费量的2%;其他包括

紧压茶、普洱茶、白茶、黄茶共10万吨,占国内茶叶总消费量的15%。

另外,从加工程度看,中国茶饮料市场自1993年起步,2001年 开始进入快速发展期。2002年,全国茶饮料的总产量接近300万吨,2009年,这一数字已超过600万吨。中国茶饮料消费市场的发展速度惊人,几乎以每年30%的速度增长,占中国饮料消费市场份额的20%。

五、影响需求的经济因素

(一)收入水平

收入是影响消费行为的最主要因素,收入水平在一定程度上决定了一国居民的茶叶消费数量和愿意支付的价格水平。随着中国城乡居民收入的增长,茶叶需求量也逐渐增加。统计资料表明,高收入家庭的消费量比低收入家庭高 1.9 倍(图 6)。

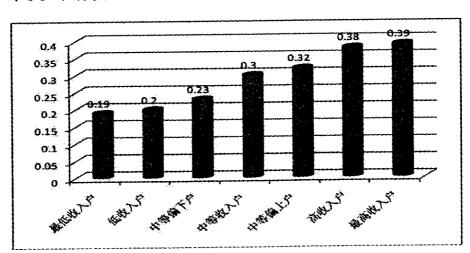


图 6 城镇居民家庭收入水平与茶叶消费量(2008年) 单位 千克注:图中数据仅限于居民直接购买量,不包含团购消费量

(二)购买力

家庭消费支出是衡量购买力的重要指标。近年来中国居民家庭消费支出持续增加,显示出了强劲的市场购买力。且随着收入的提高,中国居民的休闲保健类食品的消费支出比例也不断提高,随着饮茶有益健康研究的不断深入,中国居民对茶叶的消费必将进一步提高。

(三)产品定价及价值链

目前中国的茶叶价值链可以大致分解为鲜叶种植、初制加工、精制加工、产品批发、零售五个环节。根据我们对相关专家和企业的调查,目前中国零售价格 100 元/KG 左右的茶叶价值增值过程如图 7 所示:包括零售和批发的流通环节价值增值比例最高,可以达到 60% 左右,初制和精制加工环节增值比例约 30%,鲜叶种植环节约占 10%。调研发现,价值链上的不同利益者对产品价格的预期直接影响茶产业的可持续发展,如果渠道商从自身短期效益出发,过分压低茶叶的进货价格,会伤及供应商的生产积极性,长此以往,会影响到产品的品质。当然如果生产者过分强调生产成本投入,而不顾及渠道商的利润空间,市场开发将后劲不足。实际上,产品价格对消费者购买决策影响很大。大众消费市场的调查结果显示,60%的消费者将产品价格列为影响其购买决策的第二大主要因素。

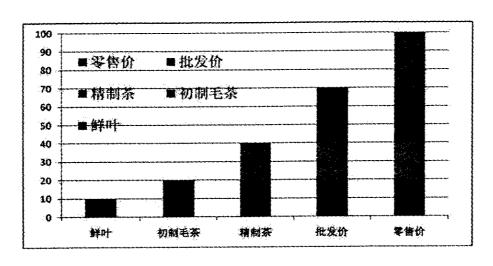


图 7 粗略估计的中国茶叶价值增值图(%)

(四)相对于其他饮料,如咖啡和其他软饮料的消费者价格

茶叶作为一种日常饮料面临着其他替代性饮料的竞争,即使茶叶的价格保持不变,替代性饮料价格上升或下降时也会使茶叶的需求量发生变化,即茶叶的需求量与其替代品的价格成同方向变化。茶是中国的传统饮品,但目前中国饮料市场中,咖啡、碳酸饮料、果汁等饮料的年消费量增长速度也很快,特别是在中国城市的年轻人群体中更是如此。这些饮料对目前中国茶叶消费构成了一定的市场竞争。但是从零售终端的价格看,茶叶还是具有很强的市场竞争力的。中国市场上咖啡成品90%是从国外进口的,需交纳进口关税,而中国国内生产的咖啡由于种植面积少总产量很低。这些因素无形中提高了包括饮料在内的咖啡成品的市场价格。在超市调查了茶、果汁、可口可乐、咖啡等软饮料的零售价格发现,茶叶价位如果采用2009年国内市场估计均价46元/千克,按常规3克冲泡一杯计算,每杯茶叶价格仅为0.138元,即使是选一12—

择价位为 1000 元/千克的龙井等名茶,每杯茶叶价格也只有 3 元, 与果汁、可口可乐和雀巢速溶咖啡价格相当。

六、非经济因素对茶叶需求的影响

(一)消费者习惯及爱好

消费者在不同历史条件下和不同的生活环境中会形成对茶叶消费的特殊偏好,一般而言茶叶消费者需求的差异性与其所处的地理环境有关,地理环境的背后实质上体现了当地的经济发达程度和传统的茶叶消费习惯,进而影响茶叶消费水平。中国地域辽阔且各地经济发展水平差距较大,所以茶叶消费的地理特征差异明显。西北、西藏茶叶人均消费量较大,其次是大中城市,南方人以饮绿茶为主,北方有很多人喜欢花茶,京、津、沪、杭等大城市的人们尤其喜欢品饮龙井茶、碧螺春等高档名优绿茶,湖南省许多人喜欢带有烟味的绿茶,福建、广东一带最喜欢品饮铁观音、水仙等乌龙茶。随着科技水平的不断提高、茶叶贮藏能力的增强,茶叶消费的季节性呈现出不明显的特征。

(二)消费者人口、年龄分布、收入分布、教育状况

人口是决定市场规模的一个重要的因素。人口高度集中地区的茶叶需求也大。人口增长快慢及其构成的变化,对茶叶消费需求具有决定性的影响。调查表明,随着教育水平,收入和年龄的增加,茶叶的消费倾向也增加。在中国,传统茶叶消费的群体中老年人占有很大比重。年轻人则更喜欢时尚、健康的快速消费品,如茶

饮料。在北京、上海、广州、武汉、成都、沈阳、西安七大城市进行的 茶饮料消费者调查中发现,茶饮料目前的消费者中以青年女性为 主,这与青年女性消费者看重茶饮料的健康、时尚特性不无关系, 另外喝茶饮料不发胖是女性多于男性选择茶饮料的主要原因之 一。

(三)质量以及对产品质量的认识

随着中国居民生活水平的不断提高,茶叶的安全意识显著增 强,要求供给的茶叶产品无污染、无农药残留的呼声越来越强烈。 为了消费安全,人们对上市茶叶的选择性普遍增强,特别是城市消 费者,其选择依据由过去的以价格为主向以安全无污染为主转变。 目前中国的茶叶消费从大宗茶为主转向以名优茶为主,导致近年 来中国名优茶呈跳跃式发展。有机茶成为近年茶消费的另一个趋 势。此外,功能性保健茶也成为茶消费的新趋势。专家指出,新兴 的保健茶在保持传统茶香的基础上,还根据不同的消费需求,加入 了诸如玫瑰、金银花、决明子等保健中药材,满足了相当一部分消 费群体的要求,具有较高的附加值。在北京的几个茶叶连锁经销 商的协助下,我们用问卷方式随机访问了300多个消费者,调查结 果显示,消费者最关注的是茶叶的品质,相比价格、品牌、服务、包 装、促销优惠等多项选择,有33%的受访者将品质放在购买茶叶 选择标准的首位,其次是产地环境和价格。有趣的是不同的消费 者判断茶叶品质的标准有所不同,高档名优茶的消费群体更注重 产品的外形,而大宗茶消费者注重茶叶的口感。

(四)消费者的知识和意识

随着人们生活水平的日益提高,不同的消费者购买茶叶的动机和目的也不尽相同。现阶段,茶叶消费呈现出个性化、保健化、礼品化、高档化等新趋势。以闽南地区茶叶消费为例,根据调查,在该地区有近四成的消费者出于保健原因购买茶叶,三成的消费者出于保健原因购买茶叶,三成的消费者出于保健原因购买茶叶时则会比较倾向于具有养颜美容和减肥功能的茶叶。调查显示,消费者对茶叶的了解程度不高,一半左右的消费群体仅是一般程度的了解。在北京市场的调查显示,消费者对茶叶关注的焦点更多还是停留在产品的物质层面或者是自身的生理需求层面,如保健、解渴或者对产品的物质层面或者是自身的生理需求层面,如保健、解渴或者社产品的物质层面或者是自身的生理需求层面,如保健、解渴或者社产品的物质层面或者是自身的生理需求层面,如保健、解渴或者社产品的物质层面对环境友好关注不多,在受访者中了解茶叶生产加工过程的人不到10%。但是,受教育程度高的消费者,对生产加工过程的关注度明显高于受教育程度低的消费群体。

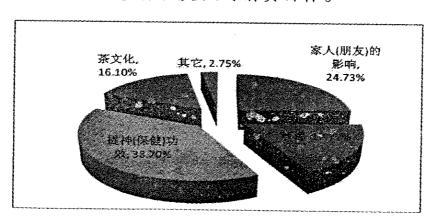


图 8 消费者选择茶饮的主要原因

七、市场开拓

(一)趋势

近年来中国茶叶消费量不断增加,今后一段时间内,国内茶叶消费仍将进一步上升。从茶类结构看,绿茶消费仍是主流,乌龙茶仍会保持增长势头,红茶消费市场开始启动,预计会进一步扩大,普洱茶市场可能会有恢复性增长。从加工程度看,深加工茶和保健茶消费会增加很快,快速消费茶特别是袋泡茶、速溶茶消费量预计会有大幅度增加。因此,中国茶产业今后的持续健康发展,是建立在重视和培育国内消费市场基础上,引导企业根据市场需要生产有自己特色的茶产品。

中国茶叶市场流通渠道的发展趋势如下:

(1)品牌连锁店经营发展较快

近年来中国主要茶叶品牌企业加快了连锁销售扩张步伐,连锁经营品牌企业在一些茶叶主要消费城市的营销网点已经十分密集,例如福建安溪八马茶叶有限公司连锁店达500多家,北京老字号吴裕泰的连锁店已经超过200家,天福集团目前在中国大陆已经开设有1012家连锁店(表5)。

表 5	中	国	大	陆	茶	叶	连	锁	밂	牌	门	店	发	展	情	况	
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公司名称	品牌	连锁总店数	销售规模(万元)
北京吴裕泰茶业公司	吴裕泰	215	53225
北京张一元茶叶公司	张一元	110	40900
天福茗茶	天福	1012	
八马茶业	八马	500	

注:张一元数据为2008年数据,吴裕泰、天福与八马茶叶为当前数据。

(2)流通主体多元化

基于价值链重构系统下的流通主体规模化、规范化、集团化,是今后流通主体的演变趋势。目前,茶叶流通参与主体正在面临以价值链重构为主题的大局面,每一参与主体只能根据自己拥有的核心能力和关键资源,从事茶叶某一环节、某一时段的业务。新的茶叶流通主体将陆续出现。如茶叶交易中介机构、冷藏保鲜中介机构、运输中介机构等等。

(3)流通要素标准化、品牌化

自2006年起,中国国内开始实施茶叶产品的市场准入许可制度,由各级质量技术监督部门组织开展审查和许可证发放(即QS认证)。这一举措的出发点是为确保茶叶质量,尤其是产品卫生质量的稳定合格性,同时也为茶叶流通的品牌化创造必要的条件。

(二)扩大市场的前景

目前中国经济保持着较高速度的发展,人民的生活水平不断提高,预计未来若干年中国经济依然会保持高速增长态势,居民收入会进一步提高(图9),茶叶消费市场预计也会随之不断扩大。

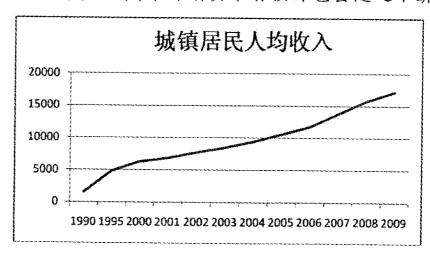


图 9 中国城镇居民人均收入变化趋势

(三)成功经验

1、弘扬茶文化,倡导茶为国饮,扩大国内茶叶消费量

上世纪90年代以来,全国各地兴起了茶文化热,各级政府尤其是茶叶主产区政府起到了积极的推动作用,组织或引导中介机构在茶叶产区和销区举办茶博会、茶叶节,大大带动了茶叶消费。政府通过倡导茶为国饮,引导消费,培养新的消费群体,为扩大市场需求做了大量有益的工作。

2、抓质量安全生产,提高产品质量,促进茶叶消费

茶叶质量是茶产业进步发展的动力和目标。中国政府高度重视茶叶产品质量安全,授权中国农业部茶叶质量监督检验测试中心每年对全国茶叶产品质量安全状况进行抽样监督检验测试;从2007年起,在茶叶行业全面实施QS制度;2006年11月1日实施的《中华人民共和国农产品质量安全法》和2009年6月1日实施的《中华人民共和国食品安全法》,为茶叶质量安全的监管提供了法律保障和依据。该法律实施后,中国政府积极推进建立茶叶质量追溯体系,当前正在全国推进标准化茶园建设。茶叶产品质量和饮用安全性的提高极大地促进了茶叶消费量增长。

3、大力宣传饮茶有益健康,带动茶叶消费量增长

近年来,中国政府不断加大对饮茶有益健康的宣传力度,引导和促进茶叶消费,并充分利用国内外关于饮茶有益于健康的研究.成果,鼓励开拓茶叶消费市场。目前茶产业已延伸到日化、餐饮、食品、医药等多个领域,饮茶有益健康理念的传播,为引导茶消费奠定了很好的市场基础,茶健康与茶文化已在大量中国消费者心

目中普及。随着中国经济的快速发展,国内茶叶消费市场还将继续扩大。

4、引导建立良性循环的市场机制

中国国内茶叶市场近年来消费量迅速增长的同时,市场价格稳步提升。这不仅取决于国民收入水平的提高和健康意识的增强,还得益于政府的市场引导。政府鼓励企业提高产品质量,通过提高茶叶质量吸引更多消费者和提高人均消费量。与大多数消费品不同,茶叶在国内的市场价格远远高于国际市场价格,2009年国内市场均价约为46元/公斤。中国国内茶叶产业已进入高质量——高价格——高消费——高效益的良性循环。

5、科技兴茶

中国政府非常注意茶叶科学研究与技术推广,每一次重大科技进步都促使了中国茶产业的跳跃性发展。通过科学研究揭示的茶叶的多种保健和治疗效应大大促进了茶叶的消费,为不断开拓茶叶市场发挥了积极的作用。

政府间茶叶工作组关于建立茶汤中农药残留最大限量的报告

I.引 言

1.2008年5月14-16日,在杭州召开的第18届IGG会议,并成立了一个新的工作组(WG):

工作组由中国作组长,印度和美国任副组长。工作组主要对下面四个方面进行工作:

- □建立茶汤中农药残留检测方法,结合农药的水溶解度和风 险评估来制定茶汤中农药最大残留限量。
 - □各成员国收集茶汤和同一样品鲜叶中农药残留量数据。
- □探讨用茶汤中的农药残留最大限量作为制定茶叶中农药残留最大限量的有关法律问题。
- □和相关国际组织代表(JMPR、CAC)进行协商,关于用茶汤中的农药残留最大限量作为制定茶叶中农药残留最大限量的有关程序
- 2.2009年5月20-22日在罗马FAO召开工作组会议,会议主要议程:
 - *建立成品茶和茶汤中农药残留检测方法;

*组织环比试验,制作绿茶和红茶中的三种农药(联苯菊酯、吡虫啉、乐果)比对试验样品,并在各成员国家和实验室进行残留量的检测试验。

II. 行 动

- 3. 通过查阅相关已经出版的文献资料,统计不同农药残留量从成品茶叶中转移到茶汤中的数据,早在1988年中国就认为茶汤中农药残留量主要取决于农药的水溶介度,第18届杭州举办的IGG会议上中国、印度和日本的实验数据已证实了这观点。
- 4.根据罗马会议的建议,建立一个有效的残留检测方法来检测成品茶和茶汤中乐果、联苯菊酯和吡虫啉农药,并计划进行环比试验,环比试验主要在8个国家11个实验室间进行,由于各种原因,只有4个国家6个实验室参加:中国(2)、印度(2)、斯里兰卡(1)和孟加拉国(1)。
- 5.根据罗马会议决定,中国(绿茶)和印度(红茶)提供检测方法,对绿茶和红茶中的不同极性的农药进行方法比较,试验结果表明,在低极性农药(联苯菊酯)的茶叶样品的测定结果用两种方法结果差异不大,然而,在高极性杀虫剂(吡虫啉和乐果)的茶叶样品的测定结果表明,结果差异相当大,干茶样必需加入一些水来提高干茶中的高极性农药的提取率。通过加水的干茶样品中的乐果和吡虫啉的农药残留量是不加水的干茶样品的几百倍以上。因此,该方法是建议加入50%的水(干茶重),该方法是在本文件附

件。结果列于表1-4。

- 6. 检测方法确立后,对实际样品进行检测验证,三个绿茶样品(喷洒农药:联苯菊酯,吡虫啉和乐果)和3个红茶样品(如上述三喷农药),2009年8月分别寄送给8个实验室。这些绿茶叶样品由中国提供(杭州,中国,2009年7月)和红茶样品由印度提供(Jorhat,印度,2009年7月)。三种农药田间喷药剂量分别如下:吡虫啉:有效成分为22.25克/公顷(印度)-45克/公顷(中国),联苯菊酯:有效成分为37.5克/公顷(中国)-80克/公顷(印度),乐果:有效成分为300克/公顷(印度)-450克/公顷(印度),兑水量为400升/公顷,喷雾器为手工操作背负式喷雾器。采样时间为新鲜的茶梢(一芽二叶)喷药后的5天(印度)和7天进行采集。
- 7. 茶汤中的农药残留量水平跟它的水溶性相关,试验结果表明:水溶解度较高的农药,在泡茶过程中的农药浸出率较高。环比试验试结果附于表 5 and 6。

皿.结论

8. 十八届罗马会议工作组制定了具体的行动计划,首先建立和完善茶汤中农药残留的检测方法,工作组建议,在 2010 - 2011年开展以下几个方面工作:

#在制定茶叶中农药残留限量标准时应该考虑茶泡中农药的 `浸出率。

#进一步通过比对试验,制定一个红茶和绿茶中农药残留检测 — 22 —

的标准方法。

#开展茶叶样品中各种农药浸出率的研究。

#通过上面各种农药浸出率的研究,对茶泡进行农药风险评估。

#进一步提出在考虑茶泡过程中农药的浸出率的基础上来制定茶叶中农药残留限量值 MRLs。

9. 在项目资金问题上,IGG 应向 CFC 或其他组织进行沟通,尽量能争取到它们在资金上的支持。

环比试验结果

目的:根据罗马会议的建议,根据中国(绿茶)和印度(红茶) 白日提供的检测方法,进行茶叶中和茶汤中乐果、联苯菊酯和吡虫 啉的残留量检测。试验方法比较列于表 1-4,茶叶和茶汤中农药 残留量结果列于表 5-7

表 1 绿茶结果(中国 实验室 1)

教药名称	成品茶(r	ng/kg)	茶汤(mg	/L)	漫出率%	
** = 9 TI 10'	方法1	方法2	方法1	方法2	方法 1	方法2
联苯菊酯	7.55	5.89	0.026	0.019	0.34	0.32
乐果	0.65	0.118	0.57	0.398	87.69	337.29
吡虫啉	4.69	0.48	2. 84	2.35	60.55	489.58

表 2 红茶结果(中国 实验室 1)

牧药名称	成品茶(1	ng/kg)	茶汤(mg	/L)	浸出率%	
ישי בדיניב	方法1	方法2	方法1	方法2	方法1	方法2
联苯菊酯	1.93	1.91	0.024	0.022	1.34	1.15
乐果	0.028	0.004	0.023	0.024	82.14	600.00
吡虫啉	0.294	0.026	0.156	0. 190	53.15	730.77

表 3 绿茶结果(中国 实验室 2)

农药名称	成品茶(r	ng/kg)	茶汤(mg	/L)	浸出率%	
**= 2 2 2 4 V	方法1	方法2	方法1	方法2	方法1	方法 2
联苯菊酯	6.798	4.11	0.120	0.110	1.77	2.68
乐果	0.695	0.116	0.563	0.546	81.0	470.69
吡虫啉	5.270	1.341	3.768	2.078	71.5	155.0

表 4 红茶结果(中国 实验室 2)

收药名称	成品茶(1	ng/kg)	茶汤(mg	/L)	漫出率%	
	方法1	方法2	方法1	方法2	方法1	方法2
联苯菊酯	1.384	1.013	0.021	0.020	1.52	1.97
乐果	0.021	0.003	0.022	0.028	104.8	933.3
吡虫啉	0. 257	0.010	0. 135	0.122	52.5	1220.0

方法1:加50%水到干茶样品中

方法2:不加水的干茶样品

表 5 结果分析(绿茶样品)

	联苯系	r di		吡虫	k		乐果			
	干茶	茶汤	浸出率	干茶	茶汤	浸出率	干茶	茶汤	漫出率 %	
中国1	7.55	0.026	0.34	4.69	2.84	60.55	0.65	0,57	87.69	
中国2	6.79	0.12	1.77	5.27	3.77	71.50	0.69	0.56	81.0	
印度1	7.49	<0.1	0. 13	5.46	3.47	63.50	0.66	0.42	63.6	
印度 2	7.31	< 0.05	0.068	4.53	2.54	56. 1	0.40	0.36	90.0	
斯里兰卡	4. 12	0.01	0. 242	ND	ND	 	2.70	0.32	11.8?	

ND:未检测出

表 6 结果分析(红茶样品)

	联苯菊酯			吡虫啉			乐果		
	干茶	茶汤	浸出率	干茶	茶汤	浸出率	干茶	茶汤	浸出率
中国1	1.93	0.024	1.24	0.294	0.156	53. 15	0.028	0.023	82, 14
中国2	1.38	0.021	1.52	0.257	0.135	52, 50	0.021	0.022	104.8
印度1	2. 15	<0.1	4.65	0.685	0.425	62.04	< 0.05	< 0.05	
印度 2	1.84	0.052	2.82	0. 239	<0.1	41.84	<0.05	<0.05	
斯里兰卡	1.670	1.352	?	ND	ND		RNR	ND	
孟加拉国	0.043	0.03	69.76	RNR	RNR	RNR	RNR	RNR	RNR

ND:未检测出 RNR:未提供结果

表7 农药水溶解度与浸出率之间的关系

农药 联苯菊酯 吡虫啉 乐果	实验室 *	浸出率 (%)	水溶解度	
	XX = *	绿茶	红茶	(L/mg)
	6 6 6	0.92(5) * * 63.26(4) 79.58(4)	0.26(5) 55.32(4) 93.47(2)	0. 1 610 25000

*联苯菊酯:中国(2个)、印度(2个)、斯里兰卡(1个) 乐果和吡虫啉:中国(2个)、印度(2个) **提供统计的实验室数量

红茶和茶汤中联苯菊酯的检测方法 - 操作规程 (印度)

1. 前言

此方法主要适用于红茶和茶汤中的联苯菊酯残留量的检测。

2. 原理

2.1. 红茶成品茶

用乙腈:水(2:1,V/V)混合溶剂提取红茶成品茶样品,通过NaCL 盐析,经弗罗里硅土柱净化,用乙醚/正已烷洗脱剂洗脱,通过GC-ECD 检测。

2.2. 茶汤

取样方法严格遵照国际标准(ISO3103,1990)。准确称量 2 克成品茶,于烧杯中加 100ml 沸水浸泡 6 分钟。之后,用布氏漏斗抽滤,滤液冷却至室温,转移至 500mL 分液漏斗中,加入 2×100mL 正已烷,液液分配,有机相经无水 NaSO4 干燥,旋转浓缩器浓缩,用正已烷定容至 10mL,待检测。茶渣干燥后,采用成品茶前处理方法,进行提取。

3. 提取

准确称取成品茶叶 20 克,加入 150mL 乙腈:水(2:1,V/V)混 — 26 —

合溶剂,在振荡器上振荡提取2个小时,抽滤转移至500mL分液漏斗中,加入200mL4% NaCl 盐溶液和60mL 正已烷,液液分配,正已烷经无水 NaSO4干燥,收集于500mL 圆底烧瓶中。

3.1. 净化

提取液旋转浓缩至干,用 10mL 正已烷溶解后,转移至 250mL 分液漏斗中,再用 5mL 正已烷洗脱圆底烧瓶并转移至分液漏斗中,加入正已烷泡和过的乙腈 30mL 进行液液分配,乙腈相经无水 NaSO4 干燥,收集于 250mL 圆底烧瓶中,在 60℃下旋转浓缩至干,用 5mL 正已烷溶解后待上柱净化,10 克 5% 弗罗里硅土柱,50mL 正已烷预淋洗柱,样品上柱,用 100mL6% 乙酸乙酯/正已烷洗脱,收集洗脱液,在 60℃下旋转浓缩至干,用正已烷定容 10mL,上 GC 检测。

仪器条件:

仪器:Perkin Elmer Clarus 500 GC

检测器:电子捕获检测器(ECD)

色谱柱:Elite-5(30m lengthx0.25mm i.dx1 µm film thickness)

气体流速

载气(氮气):5mL/min

气相温度

进样口:180℃

检测器:300℃

柱温:210℃

进样体积:0.5μL

3.2. 饱和正已烷的乙腈预处理

加入乙腈:正已烷(3:1,V/V)于125mL分液漏斗中,振荡静置分层,收集乙腈层,待用。

4. 计算:

浓度(mg/kg) = $\frac{As \ X \ C \ X \ D}{A \ std \ X \ W}$

其中:

As=样品峰面积

Astd = 标样峰面积

C=标样浓度(ppm)

D=样品稀释因子(mL)

W=样品质量

在分析中,样品和标准品等体积进样。

红茶和茶汤中吡虫啉的检测方法 - 操作规程

(印度)

1. 前言

此方法主要适用于红茶和茶汤中的吡虫啉残留量的检测。

2. 原理

2.1. 红茶成品茶

用乙腈溶剂提取红茶成品茶样品,样品浓缩后用 HPLC - DAD 检测。

2.2. 茶汤

取样方法严格遵照国际标准(ISO3103,1990)。准确称量 2 克成品茶,于烧杯中加 100ml 沸水浸泡 6 分钟。之后,用布氏漏斗抽滤,滤液冷却至室温,转移至 500mL 分液漏斗中,加入 2×100mL 二氯甲烷,液液分配,有机相经无水 NaSO₄ 干燥,旋转浓缩器浓缩,用乙腈定容至 10mL,待检测。茶渣干燥后,采用成品茶前处理方法,进行提取。

3. 提取

准确称取成品茶叶10克,加入100mL乙腈溶剂,在振荡器上.振荡提取2个小时,抽滤转移至500 mL分液漏斗中,并用50mL乙腈洗涤残渣,在85℃下旋转浓缩至干。

3.1. 净化

提取液旋转浓缩至干,用乙腈溶解后,转移玻璃柱进行柱层析,10克弗罗里硅土柱,上下两端加1cm高的无水NaSO₄,样品上柱,用100mL乙腈洗脱,收集洗脱液,在85℃下旋转浓缩至干,用乙腈定容,HPLC检测。

仪器条件:

仪器: HPLC(Agilent; Model1100)

检测器:DAD

色谱柱:ZorbaxRxC18(4.6X250mm)

流动相:乙腈:水=35:65(V/V)

流速:1.0mL/min

检测波长:270nm

进样体积:10μL

4. 计算:

浓度(mg/kg) = $\frac{\text{As } X \text{ C } X \text{ D}}{\text{A std } X \text{ W}}$

其中:

As=样品峰面积

Astd = 标样峰面积

C=标样浓度(ppm)

D=样品稀释因子(mL)

W=样品质量

在分析中,样品和标准品等体积进样。

红茶和茶汤中乐果的检测方法 - 操作规程

(印度)

1. 前言

此方法主要适用于红茶和茶汤中的乐果残留量的检测。

2. 原理

2.1. 红茶成品茶

用二氯甲烷溶剂提取红茶成品茶样品,提取液用正已烷进行液液分配,经硅胶土柱净化,用二氯甲烷洗脱,通过 GC - NPD 检测。

2.2. 茶汤

取样方法严格遵照国际标准(ISO3103,1990)。准确称量 2 克成品茶,于烧杯中加 100ml 沸水浸泡 6 分钟。之后,用布氏漏斗抽滤,滤液冷却至室温,转移至 500mL 分液漏斗中,加入 2×100mL 二氯甲烷,液液分配,有机相经无水 NaSO4干燥,旋转浓缩器浓缩,用丙酮定容至 10mL,待检测。茶渣干燥后,采用成品茶前处理方法,进行提取。

3. 提取

准确称取成品茶叶50克,加入200mL二氯甲烷溶剂,在振荡

器上振荡提取2个小时,加入1克活性碳,剧烈振荡,抽滤,并用二氯甲烷洗涤残渣,转移至500mL圆底烧瓶中,加入3滴丙二醇,旋转浓缩至2mL。

3.1. 净化

在圆底烧瓶中加入 15% 水甲醇溶液,转移至 125mL 分液漏斗中,加入 40mL 正已烷振荡 2 分钟液液萃取,静置分层,转移下层有机相至 500mL 圆底烧瓶中,重复上述一次,合并下层有机相,再用 20mL 二氯甲烷进行液液分配三次,收集二氯甲烷层,旋转浓缩至干,用正已烷溶解后,上硅胶玻璃层析柱,层析柱装 10 克硅胶,上下两端加 1cm 高的无水 NaSO₄,先用二氯甲烷预淋洗,样品上柱,先用 50mL 二氯甲烷洗脱,并弃去洗脱液,再用 200mL 二氯甲烷洗脱,收集洗脱液,旋转浓缩至干,用丙酮定容,上 GC – NPD 检测。

仪器条件:

仪器:Hewlett Packard 5890 series II 气相色谱仪

检测器:NPD 检测器

色谱柱:HP-608(30mx0.53mmx0.5μ)

气体流速

载气(氮气):12.0mL/min

气相温度

进样口:225℃

检测器:200℃

柱温:180℃

进样体积:0.5μL

4. 计算:

浓度(mg/kg) =
$$\frac{\text{As } X \text{ C } X \text{ D}}{\text{A std } X \text{ W}}$$

其中:

As=样品峰面积

Astd = 标样峰面积

C = 标样浓度(ppm)

D=样品稀释因子(mL)

W=样品质量

在分析中,样品和标准品等体积进样。

成品茶、茶汤和茶渣中联苯菊酯的残留检测方法

(中国)

1. 前处理方法

1.1 提取

- 1.1.1 成品茶:准确称取成品茶叶 10 克,加入 70mL 丙酮,浸泡过夜,过无水硫酸钠,并用 50mL 丙酮洗涤样品瓶及茶渣,收集于 500mL 圆底烧瓶中,于在 40℃水浴旋转蒸发至微干,N2 吹干,加入正已烷溶解并定容至 10mL,待净化。
- 1.1.2 茶汤:茶叶的取样方法严格遵照国际标准(ISO3103, 1990)。准确称量 2 克成品茶(品种包括绿茶、黑茶、乌龙茶和速溶茶),于烧杯中加 100ml 沸水浸泡 6 分钟。之后,用布氏漏斗抽滤,滤液冷却至室温,转移至 250mL 具塞量筒中,加入 30 克 NaCL, 150mL 乙腈,剧烈振荡 1min,静置 30min 分层,取 100mL 上层乙腈于 500mL 的圆底烧瓶中,在 40℃水浴旋转蒸发至微干,N2 吹干,用 10mL 正已烷定容,待净化。
- 1.1.3 茶残渣:抽滤过后的茶残渣,风干后,提取方法相同于成品茶。

1.2 净化

- 1.2.1 成品茶:在玻璃柱(1.0cm,id×15cm)中加入1克 Florisl (层析用,0.25mm~0.15mm,650℃ 灼烧 4h,冷却后按质量分数11%加水,混合均匀,贮存于干燥器中备用),两端加入约 2cm 高无水硫酸钠,用 15mL 正已烷预洗,并弃去流出液,下接圆底烧瓶,放入固定架上。吸取上述样品用 1mL 上柱,用 10mL 正已烷一丙酮 甲苯(445:10:45, V/V/V)洗脱小柱,收集流出液,定容至10mL,用 GC ECD 检测。
- 1.2.2 茶汤:在玻璃柱(1.0cm,id×15cm)中加入1克 Florisl, 两端加入约2cm 高无水硫酸钠,用15mL 正已烷预洗,并弃去流出液,下接圆底烧瓶,放入固定架上。用2~3mL 正已烷-丙酮-甲苯(445:10:45,V/V/V)洗脱液溶解残留物并上柱,重复数次,收集流出液,并定容至10mL,用GC-ECD检测。
 - 1.2.3 茶残渣:净化方法相同于成品茶。
 - 2. 气相色谱测定

2.1气相色谱条件:

- a) 色谱柱: DB 1701 石英毛细管柱(14% 氰丙基 苯基 甲基聚硅氧烷: 30m×0.32mm×0.25μm)。
- b) 柱箱温度程序: 初始温度 80℃保持 1 min, 以 10℃/min 升温·至 150℃, 保持 1 min, 再以 5℃/min 上升至 260℃, 保持 5 min;
 - c) 汽化室温度:260°C; 检测器温度:300°C;

- d) 进样体积:1μL;
- e)进样方式:不分流进样;
- f) 气体流量: 载气, 氮气(99.999%), 恒流模式: 流速 2mL/min; 尾吹, 氮气, 流速 20mL/min。

3. 测定

样品溶液和标准工作溶液分别进样 1μL,记录各组分的保留 时间和峰面积。根据保留时间定性,用外标法进行定量计算。

4. 结果计算

试样中被测农药残留量以质量分数 C_x 计,数值以毫克每千克 (mg/kg)表示,结果按公式(1)计算:

$$Cx = \frac{Fa \times Cs \times Qs \times Vx \times Vex}{Fs \times Qx \times Vri \times M}$$

式中:Cx:试样中x组分的含量,mg/kg;

Fa:试样中x组分的峰面积;

Cs:标样的 x 组分的浓度, mg/kg;

Qs:标样的 x 组分进样体积, LL:

Vx:试样的 x 组分定容体积, mL;

Vex:试样的提取总体积,mL;

Fs:标样中x组分的峰面积;

Qx:试样的 x 组分进样体积, µL;

Vri:试样分取体积,mL;

M:试样的质量,g;

成品茶、茶汤和茶渣中乐果的残留检测方法

(中国)

1. 前处理方法

1.1 提取

- 1.1.1 成品茶:准确称取成品茶叶5克于150mL 离心瓶中,加入25mL 纯水浸泡茶叶1小时,加入50mL 乙腈,于均质器 IKA-18均质1分钟(转速不低于10000r/min),在离心机(转速5000r/min)离心5分钟,将上清液转移至100mL 具塞量筒中,再重复上述提取一次,加入20克 NaCL于100mL 具塞量筒中,剧烈振荡1min,静置30min分层,取20mL上层乙腈于250mL的圆底烧瓶中,在40℃水浴旋转蒸发至微干,N2吹干,待净化。
- 1.1.2 茶汤:茶叶的取样方法严格遵照国际标准(ISO3103, 1990)。准确称量 2 克成品茶(品种包括绿茶、黑茶、乌龙茶和速溶茶),于烧杯中加 100ml 沸水浸泡 6 分钟。之后,用布氏漏斗抽滤,滤液冷却至室温,转移至 250mL 具塞量筒中,加入 30 克 NaCL, 150mL 乙腈,剧烈振荡 1min,静置 30min 分层,取 50mL 上层乙腈于·250mL 的圆底烧瓶中,在 40℃水浴旋转蒸发至微干,N2 吹干,待净化。

1.1.3 茶残渣:抽滤过后的茶残渣,风干后,提取方法相同于成品茶。

1.2 净化

- 1.2.1 成品茶:在 Envi Carb 固相萃取柱中加入约 2cm 高无水硫酸钠,用 10mL 乙腈 甲苯预洗固相萃取柱,弃去流出液,下接 250mL 圆底烧瓶,放入固定架上。将上述样品用 2~3mL 乙腈 甲苯溶解并转至固相萃取柱中,再用 3×2mL 乙腈 甲苯洗涤样液品,转移至柱中,用 25mL 乙腈 甲苯洗脱小柱,收集流出液,于 40℃水浴旋转蒸发至微干 N2 吹干,加入乙腈并定容至 2mL,用 GC FPD 检测。
 - 1.2.2 茶汤:净化方法同成品茶。
 - 1.2.3 茶残渣:净化方法相同于成品茶。
 - 2. 气相色谱测定
 - 2.1 气相色谱条件:
 - a)色谱柱:DB-17 石英毛细管柱(30m×0.32mm×0.25μm)。
- b) 柱箱温度程序: 初始温度 100℃保持 1min, 以 30℃/min 升温至 220℃, 保持 10min, 再以 45℃/min 上升至 250℃, 保持 5min;
 - c) 汽化室温度:220℃; 检测器温度:250℃;
 - d)进样体积:1μL;
 - e)进样方式:不分流进样;
- f) 气体流量: 载气, 氮气(99.999%), 恒流模式: 流速 2mL/ - 38 -

min; H₂:75mL/min, O₂:100mL/min, 尾吹, 氮气, 流速 20 mL/min。

3. 测定

样品溶液和标准工作溶液分别进样 1μL,记录各组分的保留 时间和峰面积。根据保留时间定性,用外标法进行定量计算。

4. 结果计算

试样中被测农药残留量以质量分数W 计,数值以毫克每千克 (mg/kg)表示,结果按公式(1)计算:试样中被测农药残留量以质量分数 C_x 计,数值以毫克每千克 (mg/kg)表示,结果按公式(1)计算:

$$Cx = \frac{Fa \times Cs \times Qs \times Vx \times Vex}{Fs \times Qx \times Vri \times M}$$

式中:Cx:试样中x组分的含量,mg/kg;

Fa:试样中x组分的峰面积;

Cs:标样的 x 组分的浓度, mg/kg;

Os:标样的 x 组分进样体积, µL;

Vx:试样的 x 组分定容体积, mL;

Vex: 试样的提取总体积, mL;

Fs:标样中x组分的峰面积;

Qx:试样的x组分进样体积, µL;

Vri:试样分取体积,mL;

M:试样的质量,g;

成品茶、茶汤和茶渣中吡虫啉的残留检测方法

(中国)

1. 前处理方法

1.1 提取

- 1.1.1 成品茶:准确称取成品茶叶 5 克于 150mL 离心瓶中,加入 25mL 纯水浸泡茶叶 1 小时,加入 50mL 乙腈,于均质器 IKA 18 均质 1 分钟(转速不低于 10000r/min),在离心机(转速 5000r/min)离心 5 分钟,将上清液转移至 100mL 具塞量筒中,再重复上述提取一次,加入 20 克 NaCL 于 100mL 具塞量筒中,剧烈振荡 1min,静置 30min 分层,取 40mL 上层乙腈于 250mL 的圆底烧瓶中,在 40℃水浴旋转蒸发至微干,N2 吹干,待净化。
- 1.1.2 茶汤:茶叶的取样方法严格遵照国际标准(ISO3103, 1990)。准确称量 2 克成品茶(品种包括绿茶、黑茶、乌龙茶和速溶茶),于烧杯中加 100ml 沸水浸泡 6 分钟。之后,用布氏漏斗抽滤,滤液冷却至室温,转移至 250mL 具塞量筒中,加入 30 克 NaCL, 150mL 乙腈,剧烈振荡 1min,静置 30min 分层,取 50mL 上层乙腈于250mL 的圆底烧瓶中,在 40℃水浴旋转蒸发至微干,N2 吹干,待净化。

1.1.3 茶残渣:抽滤过后的茶残渣,风干后,提取方法同成品茶。

1.2 净化

- 1.2.1 成品茶:在玻璃柱(1.6cm,id×25cm)中加入5克 Florisl (层析用,0.25mm~0.15mm,650℃ 灼烧 4h,冷却后按质量分数11%加水,混合均匀,贮存于干燥器中备用),两端加入约2cm 高无水硫酸钠,用20mL正已烷预洗,并弃去流出液,用20mL正已烷一丙酮(4:1,V/V)分数次溶解样品,并上柱,弃去淋洗液,再用50mL正已烷一丙酮(1:1,V/V)分数次洗涤样品瓶并上柱洗脱,收集流出液,在40℃水浴旋转蒸发至微干,N2吹干,加入乙腈,定容至2mL。用 HPLC DAD 检测。
 - 1.2.2 茶汤:净化方法相同于成品茶。
 - 1.2.3 茶残渣:净化方法相同于成品茶。

2. 液相色谱测定

2.1 液相色谱条件:

- a) 色谱柱: Aglient TC C18 柱(4.6mm×250mm,5μm)。
- b)梯度洗脱程序:流动相及梯度洗脱条件如下表:

步骤	总时间 (min)	流速 (mL/min)	A:水(0.2%磷酸) (%)	B:乙腈 (%)
0	0.00	1	90.0	10.0
1	30.00	1	40.0	60.0
2	35.00	1	90.0	10.0

c) 检测波长:270nm;柱温:35℃;进样量10µL;

3. 测定

样品溶液和标准工作溶液分别进样 1μL,记录各组分的保留 时间和峰面积。根据保留时间定性,用外标法进行定量计算。

4. 结果计算

试样中被测农药残留量以质量分数W 计,数值以毫克每千克 (mg/kg)表示,结果按公式(1)计算:试样中被测农药残留量以质量分数C、计,数值以毫克每千克 (mg/kg)表示,结果按公式(1)计算:

$$Cx = \frac{Fa \times Cs \times Qs \times Vx \times Vex}{Fs \times Qx \times Vri \times M}$$

式中:Cx:试样中x组分的含量,mg/kg;

Fa:试样中x组分的峰面积;

Cs:标样的 x 组分的浓度, mg/kg;

Qs:标样的 x 组分进样体积, µL;

Vx:试样的 x 组分定容体积,mL:

Vex:试样的提取总体积,mL;

Fs:标样中x组分的峰面积;

Qx:试样的 x 组分进样体积, uL:

Vri:试样分取体积,mL;

M:试样的质量,g:

Development of Tea Industry in China

1. Introduction

China, the hometown of tea, is the first country in the world discovered and made use of tea in history. Currently, China is the world's largest producer and consumer of tea, ranks first in world cultivation and production of tea for many years, predominating in the world tea industry. Tea sectors employ over 80 million workers directly or indirectly in China's twenty tea belts. Tea is one of the most important cash crops in China, tea industry play a quite important role in accelerating economical development and making farmers rich in tea-planting regions. Following analysis detailed the development of tea industry in China in recent years.

2 Production, domestic supply, and international trade

2.1 Production: cultivation areas, harvest areas and yield

In 2009, tea planting area and harvest areas covered 1.867 million hectares and 1.38 million hectares, rising by 6.9% and 6.6% from 2008, respectively. The total production reached 1.35 million tones and production value amounted to 41.367 billion RMB, increasing by 16.9%. In recent years, China's tea production area, yield and value show rapid growth trends on the whole (Fig. 1).

China produces different kinds of tea, but the main is green tea. In 2008, yield of green tea, oolong tea and black tea were 92.7 million tons, 144,000 tons and 69,700 tons, accounting for 74%, 11% and 6%, respectively. Fig. 2 shows: oolong tea is unceasingly increasing; black tea decreased from 1998 to 2003, but rose since 2004; compressed tea is relatively stable in recent years; other kinds of tea also rose rapidly, meeting various needs of consumer.

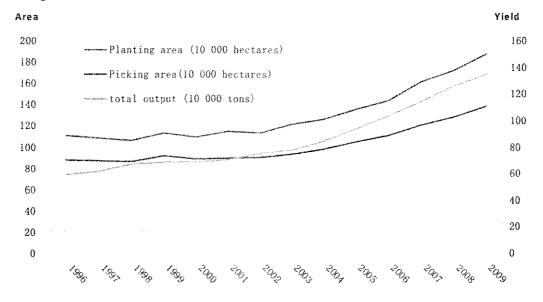


Figure 1 Trends of tea production area and yield in China

2.2 Trade: Export / re-export and import

In recent years, the amount of tea exports grew steadily in China, in which green tea, oolong tea and white tea increased, while black tea, dark tea and other special teas decreased (Fig. 3). In 2009, China exported of 303,000 tons of tea that worth \$705 million (Table 1), in which green tea were 229.3 thousand tons and 5.25 hundred million dollars; black tea 40 thousand tons and 64.37million dollars; oolong tea 24 thousand tons and 66.86 million dollars; scented tea 5.914 thousand tons and 29.63 million dollars.

Meanwhile imports of tea grew quickly. In 2008, the trade amount of tea was 302,300 tons in China, of which 5,398 tons for imports. Most of them were black tea, reaching 4,306 tons and accounting for 79.77%, while green tea was 944 tons, accounting for 17.49%.

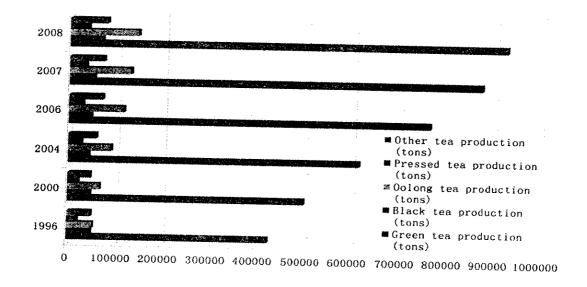


Figure 2 Structure of Chinese tea products

2.3 Consumption

China is traditionally a major tea consumer. In recent years, as the income of inhabitants increasing, the increasing rate of the annual consumption of tea stood at 10-15%, especially in main market areas. A survey showed that consumption of tea per head reached 2.50 kg annually in Guangzhou province. The total consumption of tea in 2009 is estimated to reach about 100 million tons.

Table 1 Tea Export & Import of China (10000 tonnes, ×108 US\$)

Year	Export Quantity	Export Value	Import Quantity	Import Value
1999	19.96	3.38	0.19	0.03
2000	22.77	3.47	0.24	0.04
2001	24.97	3.42	0.17	0.03
2002	25.23	3.32	0.17	0.03
2003	25.99	3.67	0.29	0.04
2004	28.02	4.37	0.23	0.06
2005	28.66	4.84	0.28	0.07
2006	28.66	5.47	0.32	0.08
2007	28.95	6.08	0.53	0.13
2008	29.69	6.82	0.54	0.18
2009	30.3	7.05		

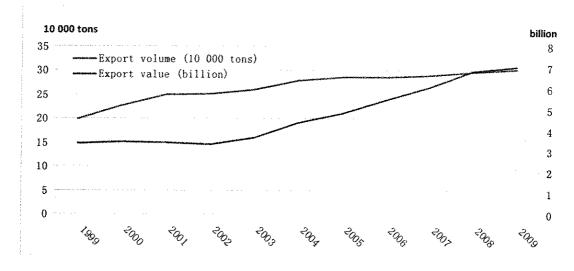


Figure 3 Trends of tea exports of China

3. Marketing channels

3.1 Introduction

From mid-1980s, the tea circulation system in China has been reformed radically. The Fig.4 shows the basal tea marketing channels existing in China at present. The Scented Tea and Border-sale Tea flow into the market after special refining process, while famous and high-quality tea enter the market directly after processed by farmers or tea plantations. Organizations that link producers to consumers mainly included nets of the mass markets, tea trade markets, specialized dealers and shops, but a part of tea o get to the consumers directly from the farmers and others should pass from mid-economy organizations to consumers. The main channel of trading famous and high quality tea in China is tea trade market, which normally contain producers, mid-organizations and tea houses.

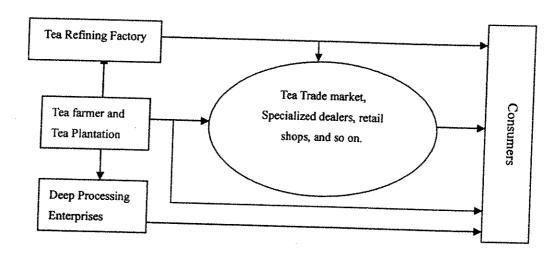




Figure 4 Current distribution channels of tea in the domestic market of China

3.2 Distribution channels of tea farmers

According to a survey carried out by China Tea Industry Economic Research Institution (CTIERI) which covered 518 tea farmers distributing in 10 important tea-producing provinces, 46.3% of them sold their tea to local leading enterprises. Some farmers chose mid-businessmen as the only or one of their production accepters, which occupy 20.3% and 26.1% correspondingly. Others sold their tea by themselves through Farm Product Markets or Specialized Cooperatives, but these only take few proportions.

Table 2 Distribution channels of tea farmers

Marketing channels	Number	Proportion
①Farm Product Markets Selling	61	11.8%
②TO Mid-businessman	105	20.3%
③To Leading Enterprises	240	46.3%
Specialized Cooperatives Selling	33	6.4%
⑤Own Shop selling	15	2.9%
Contain ①	95	18.3%
Contain ②	135	26.1%
Contain ⑤	26	5.0%
Contain ③	270	52.1%
Contain ④	64	
Contain ①②	215	12.4% 41.5%

3.3 Distribution channels of tea enterprises

A study of the CTIERI in 2009 suggests that there are two Outstanding Characteristic of choosing marketing channels for tea enterprises. One is that 74% of enterprises in the study apt to establish their own distribution channels and nets. The other is multi-channels distribution that prevails for 65% enterprises. Preliminary

analysis indicates that Profit distribution and business concepts are the main causes leading this situation. Currently, it is considered that the most profitable opportunities lie in retail links. As a consequence, to achieving more profits, more enterprises prefer sales terminal controlling through contribution of their own channels or other ways.

Table 3 Distribution channels of tea enterprises

Channels	Number	Proportion	
①Own Sales Networks	45	22%	
②by supermarket	1	0%	
③consignment sales	6	3%	
Wholesale market	4	2%	
Swaiting for consumers	10	5%	
Two ways	58	29%	
Over three ways	74	36%	
Contain ①	151	74%	
Contain ②	66	33%	
Contain ③	83	41%	
Contain ⑤	81	40%	

4. The Analysis of Market Share

4.1 Channels of Wholesale and Retail

The market shares of tea in China are mainly on domestic, which accounts for 77.8% of sales and the broad market 22.2%. In domestic market, tea trades endure the process from wholesale to retail. And wholesale markets have two types; original and selling sites. Presently, there are comparatively few of original wholesale market in China except those counties with high yield in tea. However, the wholesale markets in selling sites are spread almost every large and medium-size cities, there are large scales of tea wholesale market in those metropolises like Beijing, Shanghai, Tianjin, Guangzhou, Wuhan and Shenyang, in which peddling can also be found. The retail distribution channels in China's tea market are major in tea special stores and original trade markets. The reason is that in these sites tea consumers can taste various kinds of commodity in different prices, favors and colors, such a great selective space can satisfy different preferences. Comparing with consumers' disability of tasting or bargaining in supermarket and emporium, in tea original sites, they can find out their favorable tea with proper price through negotiation with sellers depending on the benefit of huge tea supply and fierce competition among sellers, the full-scale and transparent market information in those sites. Consumption in tea room, customers care more about casual experience, therefore their comfortable environment and service of high added-value make tea prices higher than other places. According to investigations, customers from tea original and selling sites choose dissimilar selling channels. In origin, customers prefer to purchase tea with tea farmers. They can make their final decisions through their own judgments of tea quality after on-the-spot experience, by the inspection of tea garden environment and tea productions. While customers in selling market sites prefer the tea special stores with the purpose of

convenience and quality guarantee. With the quick expanding of brand chains, tea trade becomes more and more branding and converging. The improvement of market information and logistics thrives the wholesale market in origin, therefore some large tea-selling enterprises purchase directly from those sites, some of them even build their own production bases in those sites to reduce the links of raw material trade and their production and operation costs.

4.2 The Import Share Apply for Original Rules

Chinese tea is welcomed by consumers all over the world. By the end of 2009, Chinese tea has already exported to more than 110 countries and regions. The import amount of the top eight countries among those countries took up 60% of Chinese tea export. These countries and regions like Morocco, America, Republic of Uzbekistan, Japan, Pakistan, Algeria, Mali, Nigeria, Senegal, Tunis and Hong Kong are the main Chinese green tea importers; Our black tea mostly is exported to Russia, America, U.K., Hong Kong and Pakistan; special tea (oolong tea and scented tea) are mainly exported to Japan. The chief importers of Chinese tea are Morocco, Japan, Republic of Uzbekistan, America and Russia. Recent years, Morocco has always been the largest importing country of Chinese tea and reached 58.5 thousand tons in 2009; Republic of Uzbekistan is the second and 22 thousand tons. Russia became the third largest importing country of Chinese tea and reached 21 thousand tons; due to the influence of financial crisis, America are the fourth largest with a volume of 19 thousand tons; Japan continues to decrease the importing of Chinese tea and less than 19 thousand tons in 2009; Pakistan imported 14.8 thousand tons of Chinese tea in 2009. (fig.5)

Importing amount of Chinese tea (ton) 70000 60000 20005 50000 20000 30000 20000 10000 0 Morocco Japan Uzbekistan America Russia

Figure 5 Changes of importing amount of Chinese tea in main importing countries

4.3 Product Category of Consumption

In china, people tend to consume green tea more, the consumption of it in 2006 was 385 thousand tons, which made up 58% of total domestic consumption; the

amount of oolong tea and scented tea came closely, each of them are 80 thousand tons respectively, which occupy 12% of total home consumption; the volume of black tea consumption is 15 thousand tons and accounts for 2%; the other kinds of tea such as brick tea, puer tea, white tea and yellow tea were 100 thousand tons and take the proportion of 15.

In addition, China established tea beverage market in 1993, in 2001 this market turned into a flourish time. The total output of domestic tea beverage in 2002 nearly reached 3 million tons, in 2009 it rose to more than 6 million tons. The Chinese tea beverage market develops rapidly and almost increases by 30% each year, for which account 20% of Chin's beverage market.

5. Economic Factors Affecting Demands

5.1 The Levels of Incomes

Income is the major force that affects tea consumption activities, as to the residents of one country, the consumption volume and desirable price of tea depend on their levels of personal incomes. With the growing incomes of urban and rural residents, tea demands increase steadily. Statistical references indicated that the tea consumption volume of high-income households is more than 1.9 times of that of low-income households (fig. 6).

Per capita tea consumption (Kg)

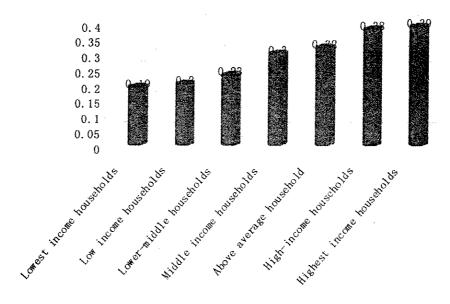


Figure 6 Relation between urban household incomes and tea consumption in 2008

Note: The data is limited to the residents' direct purchases, does not include the group's consumption.

5.2 The Purchasing Power

Household's living expenses are the most important implication of purchasing

power. The recent years observed strong increase advising a forceful purchasing power for tea. Accompany with higher income, the proportion of living expenses for leisure and health protection foods improves. The more researches of the benefit of drinking tea for physical health, the more Chinese residents will consume the commodity of tea.

5.3 Setting Price of Production and Its Value Chains

Presently, the value chains of Chinese tea can be divided into five links: the growing of tea plant, the primary processing, the refining processing, the wholesale of production and retail. According to our investigations from relating specialists and enterprises, the process of value-added of commodity tea with the retailing price of 100 yuan (RMB) in China are properly expressed in Fig. 7, as we can see, the distribution including retail and wholesale has the highest proportion of value-added and reaches 60% of final value; the process of primary and refine production add by 30%; tea plant growth account for 10% of value adding. In addition, businessmen with different interests in this value-chain can affect the sustainable development of tea industry by their expecting of tea prices. For instance, if the middlemen thinking of their own short term benefit bring down the price too much to dampen manufacture's enthusiasm to produce, and finally the quality of tea will be undermined. On the other hand, manufactures' over-emphasis on costs of production and disregarding of middlemen's interest space will make further market exploration harder. Actually, product price plays an important role in consumers' decision on purchasing. The survey from the mass consume market displays that about 60% of consumers consider product price as the second important factor that affect their purchasing decision.

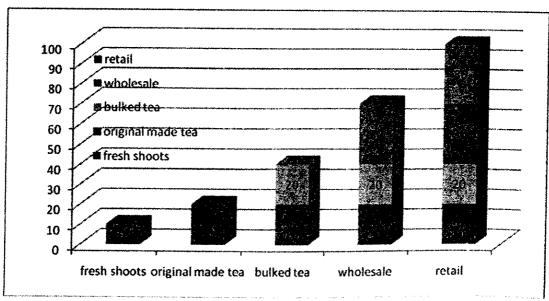


Figure 7 Estimated price changes from raw material to commodities (%) (From left to the right: fresh shoots, original made tea, bulked tea, wholesale and retail).

5.4 The Comparison of Consumer Price between Tea and Other Kinds of Soft Drinks such as Coffee

Tea is considered to be one of usual drinks that faces competition from other alternative beverages. The fallings and risings of alternative beverage prices will make an influence on tea demand even though the price of tea unchanged, it means that changes of alternative beverage price make the occurrence of a change of tea demand in the same direction. As Chinese traditional drink, tea meets with a fierce market competition from other thriving beverage with rapid consumption increasing such as coffee, carbonated drink and juice, when young generation in Chinese urban cities especially fond of these alternative drinks. Nevertheless, suggested from its final retailing price of value chain, tea still owes the forceful market competition. In view of domestic production of coffee with small planting area and low volume, about 90% of them are imported from other countries, which virtually improve market price of these beverages like coffee. A market retailing price survey of soft drinks as tea, juice, coke and coffee indicates that the cost of a cup of tea used the article of the average price 46 yuan (RMB) per kilogram in 2009 domestic market and conventional brewing amount of three grams each cup is just 0.138 yuan (RMB); the cost of tea of famous and quality like Longjing is just 3 yuan (RMB), which are equal to cost of juice, coke and instant coffee.

- 6. The impact on demands of tea for non-economic factors
- 6.1 The consumer's personal costume and hobby

Geographic position decides the local level of economic development and consumption habit of tea, and also impact tea consumption. Thus, consumers have different interests on tea consumption according to different position. There is an obviously geographic character of tea consumption in china, which has a vast territory and a great difference of economic level around.

People in the north of china prefer scented tea, while southerners prefer green tea. And the consumption of western minority is dark tea mainly. People in Beijing-Tianjin and Shanghai-Nanjing-Hangzhou drink high grade green tea like Longjing tea and Biluochun tea. People from Hunan prefer green tea have smoky flavor, and people from Guangdong-Fujian like oolong tea best.

6.2 Population Characteristics

Population is a so important factor that would decide the market size, and demand of tea is greater in populated areas. Rate of population growth and changing of demographics have a decisive impact on consumption demand of tea. The survey showed that age also plays an important role on tea demand. The elder consumers take a larger proportion in traditional tea consumption groups. Young people prefer FMCG, something faddish and healthy, just as tea beverage. The survey, which focused on tea beverage consumption in Beijing, Shanghai, Guangzhou, Wuhan, Chengdu, Shenyang and Xi'an, showed that tea beverage consumption is mainly based on young women, choosing tea beverage for they are faddish and healthy, and drinking tea beverage can make their weight reducing.

6.3 Consumer consciousness

With the elevation of living standard in China, public awareness and demand for safer tea has risen significantly, they shit consumption concept from price-based to safety-based, and consumption of organic tea is rising. Functional healthy tea becomes a new trend. some experts point out that, in order to satisfy various consumption demands on the base of keeping traditional smells of tea, new health-care tea contains some traditional Chinese medicinal materials such as rose, honeysuckle and semen cassiae torae and have rather high added-value.

A questionnaire investigation was implemented randomly by more than 300 people with the help of several train dealers on tea in Beijing. And the result showed that consumers are mostly concerned on tea quality among price, brand, service, packing, discount, producing area and quality. 33% respondents put quality in the first place, when buying tea, and then are producing area and price. And people got quite different standards; grade tea consumers pay more attention to the appearance of tea, while bulk tea consumers pay their attention to the taste of tea.

At present stage, tea consumption turns into a new trend of personality, health caring, gifting and costliness. A case study of southern Fujian showed about 40 percent consumer buy tea for health, and 30 percent for gift, female consumers for skincare and slimming function of tea. Few consumers know tea quality quite well in China even in Guangzhou, where the tea culture atmosphere is denser than other regions, just 28.3% of them well understand the knowledge of tea and about half of them know the general information of tea. An investigation from Beijing market suggested, consumers pay more attention to the material levels of tea or their physical demands on tea such as keeping fit, quenching thirst or influence by other individuals. Less than 10% of them familiar with the procedure of tea, few consumers care about the planting and processing of tea and their environment-friendly procedure while producing.

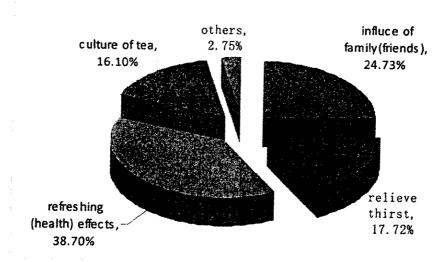


Figure 8 Main reasons for drinking tea by consumers

In recent years, consumption of Chinese tea is increasing. For some time, domestic tea consumption will further increase. From the structure of tea, green tea consumption is still mainstream, oolong tea will maintain growth, black tea consumption market has begun and is expected to expand. There may be restorative puer tea market growth. Viewed from the degree of processing, deep-processed tea and health protection tea consumption will increase rapidly, and fast consumption teas, especially tea bags and instant tea consumption are expected to be greatly improved. Therefore, to further promote continuous and healthy development of China's tea industry, the direction of trade should be adjusted, and the domestic consumer market should be paid more attention to and be preferably nurtured, enterprises must choose right target market according to the characteristics of their products, formulate marketing strategies suitable for the local market.

The distribution trend of China's tea market are as follows:

(1) Rapid development of brand chain store

China's major tea brands in recent years have accelerated the pace of expansion of chain sales, the major chain brands in some tea consumption major cities have very intensive marketing outlets. For example, Fujian Anxi Bama Tea Co. opens up to 500 chain stores; Beijing Wu Yu Tai, a traditional and famous shop, has more than 200 chain stores; Tenfu Group has 1,012 chain stores (Table 5) in mainland China.

Table 5 The development of tea brand's chain stores in Chinese Mainland

Company Name	Brand	Number of chain	Selling-scale
		store	(million)
Beijing Wu Yu Tai Tea	Wuyutai	215	53,225
Company			
Beijing Zhang Yi Yuan	ZhangYiYuan	110	40,900
Tea Company			
Tenfu's Tea	Tenfu	1,012	
Bama Tea	Bama	500	

Note: ZhangYi Yuan's data for the 2008 data, the data of Wu Yu Tai, Tenfu's Tea and Bma Tea for the current data.

(2) Diversification of current principal part

Scale, standardized and grouping of current principal part which are based on Reconstruction systems of the value chain are the trend of current principal part. Currently, Tea current principal part is facing the situation of the main theme of reconstruction. Each principal part must be in accordance with their own core competencies and key resources, participating some links in the tea or a period of business. The new current principal part will be expected successively, such as the tea trade intermediaries (such as tea brokers, etc.), cold storage agency, transportation intermediaries and so on.

(3) Standardization and branding of circulation elements

Since 2006, China launched the market access permit system of tea products and

review and licensing(ie, QS Certification) implemented by the technical supervision departments at all levels. The starting point of this initiative is to ensure the quality of tea, especially the stable eligibility of product quality, meanwhile, to provide the necessary protection of tea brand in circulation.

(B) Prospects of expanding the market

At present, China's economy maintains a high rate of development and people's living standards improves continuously. Therefore, in the next few years, China's economy is still expected maintaining rapid growth, and people's income will further increase (Fig. 9), and simultaneously, tea consumption market is expected to continue expansion.

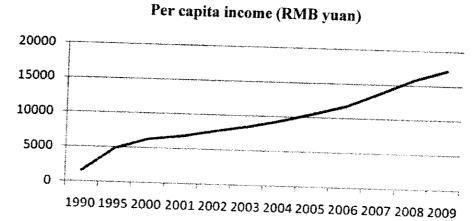


Figure 9 Trends of per capita income of Chinese urban residents (C) Experience of success

1. Promoting the tea culture, and advocating tea for the national drink, expanding domestic tea consumption

Since 90s of last century, the hot tea culture rise across the nation, tea at all levels of government particularly in the main producing areas, played a positive role in organizing and leading intermediaries to hold tea expo and tea festival in the tea-producing areas and marketing areas, which greatly promoted tea consumption. Government, by way of advocating for the national drink of tea, guiding consumption and developing new consumer groups, has done a lot of useful work in expanding the market demand.

2. Focusing on quality and safety of production, improving product quality, promoting tea consumption

Government, through controlling quality safety to satisfy consumer demand, has promoted the growth of tea consumption. Since 2007, under the full implementation of the QS system in the industry, and now Government's strong supervision, quality safety becomes the industry-wide consensus. The Government, by encouraging enterprises to increase the control tea source and the reform of processing environment, tea quality is greatly improved. For example, Zhejiang Province, since the second half of 2006, through government subsidies, the number of tea factories reformed in the province have been close to 500; 123 companies have certified by the Pollution-free Production Bases;109 tea products have identified by the Green Agricultural Products; nearly 20 products have certified by Green Food Certification;

94 600 acres of organic tea garden of 177 enterprises have passed the domestic or international organic tea certification; more than 20 enterprises have passed the ISO9001 or ISO14000 certification.

Tea quality is the driving force and goals for progress and development of tea industry. In recent years the Chinese government attaches great importance to tea product quality safety. Tea Quality Supervision and Testing Center of Chinese Agricultural Ministry, carries on the spot-check to quality and safety of tea product each year. The implementation of "Law of Quality Safety of Agricultural Products in People's Republic of China" in November 1, 2006 and the implementation of "Law of Food Safety in The People's Republic of China" in June 1, 2009, provide protection and legal basis of tea quality safety regulation. After the implementation of the law, Chinese Government actively promotes the establishment of tea quality tracing system and is currently promoting the standardization of tea all over the country. The improvement of tea product quality and drinking safety greatly promoted the growth of tea consumption.

3. Vigorously propagandizing health benefits of tea drinking, thereby driving tea consumption growth

In recent years, Chinese government has continued to increase propaganda of health benefits of tea drinking, and guides and promotes tea consumption. Besides, Chinese government makes full use of foreign research results on healthy of tea drinking, and encourages the development of new markets for tea consumption. Consequently, at present, tea industry has been extended to daily chemical, catering, food, medicine and other areas. The spread of health benefits of tea drinking ideas has laid a good market base for guiding tea consumption. Tea Health and Tea Culture have been popular in a large number of consumers' minds in China. With China's rapid economic development, domestic tea consumption market will continue to expand.

4. Guiding the establishment of a virtuous circle of market mechanisms

China's domestic tea consumption in recent years, has grew rapidly, and market prices has increased steadily. This not only depended on increases of income levels and health awareness, but also benefited from the government' guide on the market. The Government encourages enterprises to improve product quality and attract more customers and increase per capita consumption by improving the quality of tea products. Unlike most consumption goods, the market price of tea in China is much higher than international market price, the average price is about 46 yuan (RMB) / kg in domestic market in 2009. China's domestic tea industry has access to a virtuous circle of high quality, high price, high cost and high efficiency.

5. Science and technology of tea

Chinese government pays great attention to scientific research and technology generalization of tea. Each major scientific and technological advance has contributed to the jump development of China's tea industry. A variety of health and treatment effects of tea, revealed by scientific research, contribute significantly to the consumption of tea and play an active role for developing tea market.

REPORT OF THE WORKING GROUP ON THE DEVELOPMENT ON ESTABLISHING MRLs IN TEA ON THE BREW

I. INTRODUCTION

1. At the Eighteenth Session of the IGG on Tea, which was held in Hangzhou, China in 2008, there was agreement to establish a new Working Group (WG). On the para 27, Report of 18th Session of IGG on Tea, Hangzhou 14-16 May, 2008:

Sampling of tea on the brew would be investigated by the WG under the leadership of Prof. Chen (China) with the assistance of Dr Chaudhuri and Mr Simrany (USA). This Working Group would:

- □ Combine the existing methodology of measuring residues in the tea brew, risk assessment and solubility of chemicals for establishing MRLs.
 □ Collect data on tea brew as well as leaf for the same sample
 □ Investigate the legal considerations for establishing MRLs by this method;
 □ Consult with the related international agencies, e.g. JMPR, Codex Alimentarius and
- 2. The Meeting of the Working Groups was conducted at FAO in Rome from 20 to
 - *Establish the methodology for tea brew and dry tea;

22 May 2009, The Meeting instructed the WG to:

*Organizing the ring tests of green tea and black tea samples from tea garden sprayed with three pesticides (Bifenthrin, Imidacloprid and Dimethoate)in different countries and laboratories:

II. ACTIVITIES OF THE WG

- 3. Compilation of literature published on transfer rate of pesticide residue from made tea to tea brew showed a wide difference of pesticide residue in tea infusion which is depended largely by the water solubility of pesticide as early as in 1988 in China. The experimental data on the investigation from China, India and Japan ere presented at the 18th IGG at Hangzhou, China.
- 4. According to the recommendation in the Rome Meeting, a validation of established methodology for determining the residues of dimethoate, bifenthrin and imidacloprid in the tea brew was planned to conduct through a ring test. The ring test are planned to conduct in 11 labs in eight countries. Due to the various reasons, the analysis was conducted in six labs of four countries: China (2), India (2), Sri Lanka (1) and Bangladesh(1)

national authorities.

- 5. As decided in the Rome Meeting, China recommended the method for green tea, and India recommended the method for black tea. A comparative determination of both methods was conducted with green tea and black tea samples fortified with three pesticides in different polarity. Investigation showed that the results of determination on the tea samples with low polarity pesticide (Bifenthrin) by two methods are similar, however, the results of determination on the tea samples with higher polarity pesticides (Imidacloprid and Dimethoate) showed a quite greater differences. Adding some water to the dry tea sample is necessary for improving the extractive rate of pesticides with high polarity from dry tea. If no water added to the dry tea, the residue level of these pesticides with high polarity in dry tea samples will several hundreds times lower than those in tea brew. So, the method is recommended to add 50% water (weight of dry tea) to the tea sample. The method is attached in this document. The results are listed in Table 1-4.
- 6. After the validation of the established methodology, three green tea samples (sprayed with three pesticides accordingly: Bifenthrin, Imidacloprid and Dimethoate) and three black tea samples (sprayed three pesticides as above) were sent to 8 labs at August of 2009. These tea samples were provided d by China (Green tea, TRI, Hangzhou, China, July 2009)) and India (Black tea, TRA, Jorhat, India, July 2009). The dosage was imidacloprid 22.25 g a.i. ha⁻¹ (India) –45 g.a.i. ha⁻¹ (China), bifenthrin 37.5 g a.i. ha⁻¹ (China) –80 g a.i. ha⁻¹ (India), dimethoate 300 g a.i. ha⁻¹ (India) 450 g a.i. ha⁻¹ (India), Spray volume 400 L/ha, Sprayer: Hand operated knapsack sprayer. The fresh tea shoots (one bud and two leaves) from treated tea plants was harvested at 5 days (India) or 7 days after spraying.
- 7. The residue level of pesticide in the tea brew is closely related with the water solubility of pesticide. The higher water solubility of pesticide showed higher extractive rate of pesticide during the brewing process from made tea. The results of ring test is attached in Table 5 and 6.

III. CONCLUSION

- 8. The Action Plan agreed at the Eighteenth Session and the Working Group Meeting in Rome is being progressed. The methodology for determining the residue in tea brew was established and amended. The Working Group recommended to investigate the following aspects in 2010-2011.
- # Legal consideration for establishing MRLs of various pesticides in tea by considering the situation during brewing process
- # To establish one or two common methods for the determination of black tea and green tea after a further ring test
- # To conduct the experiment on the transfer rate of various pesticides used in tea production
- # To conduct the risk assessment of pesticide in made tea to drinker during the brewing process according to the above investigation on the transfer rate of various

pesticides

To put forward a suggestion on how to establish the MRL of pesticide in made tea in considering the transfer rate of pesticide from made to tea brew for discussion.

9. The funding problem should be considered and identified. It is recommended that IGG should contact with CFC or other organization for getting the supporting funds.

Enclosure I RING TEST FOR METHODOLOGY

Objective: According to the recommendation in the Rome Meeting, a validation of established methodology for determining the residues of dimethoate, bifenthrin and imidacloprid in the tea brew was planned to conduct through a ring test. The Black tea method is provided by India, the green tea method is provided by China. The comparative results of methodology are listed in Table 1-4. The results of tea samples are listed in Table 5-7.

Table 1 Result of Green Tea (China Lab 1)

Pesticide	Made tea(mg/kg)		Tea infus	ion(mg/L)	Brewing rate%		
Name	Method 1	Method 2	Method 1	Method 2	Method 1	Method 2	
Bifenthrin	7.55	5.89	0.026	0.019	0.34	0.32	
Dimethoate	0.65	0.118	0.57	0.398	87.69	337.29	
Imidacloprid	4.69	0.48	2.84	2.35	60.55	489.58	

Table 2 Result of Black Tea (China Lab 1)

Pesticide	Made tea(mg/kg)		Tea infus	ion(mg/L)	Brewing rate%		
Name	Method 1	Method 2	Method 1	Method 2	Method 1	Method 2	
Bifenthrin	1.93	1.91	0.024	0.022	1.34	1.15	
Dimethoate	0.028	0.004	0.023	0.024	82.14	600.00	
Imidacloprid	0.294	0.026	0.156	0.190	53.15	730.77	

Table 3 Result of Green Tea (China Lab 2)

Pesticide	Made te	a(mg/kg)	Tea infus	ion(mg/L)	Brewing rate%		
Name	Method 1	Method 2	Method 1	Method 2	Method 1	Method 2	
Bifenthrin	6.798	4.11	0.120	0.110	1.77	2.68	
Dimethoate	0.695	٠ 0.116	0.563	0.546	81.0	470.69	
Imidacloprid	5.270	1.341	3.768	2.078	71.5	155.0	

Table 4 Result of Black Tea (China Lab 2)

Pesticide	Made te	a(mg/kg)	Tea infus	ion(mg/L)	Brewing rate%		
Name	Method 1	Method 2	Method 1	Method 2	Method 1	Method 2	
Bifenthrin	1.384	1.013	0.021	0.020	1.52	1.97	
Dimethoate	0.021	0.003	0.022	0.028	104.8	933.3	
Imidacloprid	0.257	0.010	0.135	0.122	52.5	1220.0	

Method 1:50% water is added to the dry tea sample.

Method 2: No water is added to the dry tea sample.

Table 5 Results of analysis (Green tea sample)

	Bifenthrin		Imic	Imidacloprid			Dimethoate		
	Dry tea	Tea Brew	Extractive	Dry tea	Tea Brew	Extractive rate %	Dry tea	Tea Brew	Extractive
China 1	7.55	0.026	0.34	4.69	2.84	60.55	0.65	0.57	87.69
China 2	6.79	0.12	1.77	5.27	3.77	71.50	0.69	0.56	81.0
India 1	7.49	<0.1	0.13	5.46	3.47	63.50	0.66	0.42	63.6
India 2	7.31	<0.05	0.068	4.53	2.54	56.1	0.40	0.36	90.0
Sri Lanka	4.12	0.01	0.242	ND	ND		2.70	0.32	11.8

ND: not detected

Table 6 Results of analysis (Black tea sample)

	Bifenthrin			Imidacloprid			Dimethoate		
	Dry tea	Tea Brew	Extractive	Dry tea	Tea Brew	Extrac- tive rate %	Dry tea	Tea Brew	Extractive
China 1	1.93	0.024	1.24	0.294	0.156	53.15	0.028	0.023	82.14
China 2	1.38	0.021	1,52	0.257	0.135	52.50	0.021	0.022	104.8
India 1	2.15	<0.1	4.65	0.685	0.425	62.04	<0.05	<0.05	AP-LIT-US
India 2	1.84	0.052	2.82	0.239	<0.1	41.84	<0.05	<0.05	****
SriLanka	1.670	1.352	?	ND	ND	**************************************	RNR	ND	ever-sa
Bangladesh	0.043	0.03	69.76	RNR	RNR	RNR	RNR	RNR	RNR

RNR: result not reported

ND: not detected

Table7 Relationship between the water solubility of tested pesticides and the extractive rate of pesticide from made tea to tea brew

Pesticide	Numbers of Lab*	Extractive pesticide resimade tea to (%)	Water solubility (L/ mg)	
		Green tea	Black tea	
Bifenthrin	5	0.92	0.26	0.1
Imidacloprid	4	63.26	55.32	610
Dimethoate	4	79.58	97.31	25000

^{*}Information of Bifenthrin reported from two labs from China, two labs from India and one lab from SriLanka.

Information of Imidacloprid and Dimethoate reported from two labs from China, two labs from India.

Enclosure II

SOP for the determination of Bifenthrin residues in Black tea and tea brew (provided by India)

1. Introduction

This method is applicable for the determination of bifenthrin residue in black tea and tea infusion.

2. Principle

2.1. Black tea

Bifenthrin is extracted with acetonitrile: water (2:1, v/v) mixture from the black tea samples. The extract is partitioned with sodium chloride and cleaned up through florisil column. Then it is eluted with ether/hexane mixture. The concentrated residue in the eluate is determined by GC with electron capture detector.

2.2. Tea brew

Two gram of made tea was infused in 100 mL of boiling water (ISO 3103-1990). After 6 min of brewing, the water extract was filtered, cooled and partitioned with 100 mL of hexane (twice). The organic phase was passed through anhydrous sodium sulphate. The extract was concentrated by evaporating in a rotary vacuum evaporator and diluted with 10 mL hexane and analysed for the residues of bifenthrin. The spent leaves were dried between the folds of filter paper and residues were extracted following the method described above for black tea.

3. Extraction

Twenty gram of black tea sample is extracted with 150 mL of acetonitrile: water (2:1, v/v) by shaking it in a mechanical shaker for two hours. The contents are filtered and to the filtrate 200 mL of 4% NaCl and 60 mL of hexane are added. After partitioning, the hexane layer is passed through anhydrous sodium sulphate layer to a 500 mL round bottomed flask.

3.1. Clean up

The extract is evaporated to dryness on a rotary vacuum evaporator and the residue is dissolved in 10 mL hexane and again transferred to 125 mL separating funnel. The round bottomed flask is rinsed with 5 mL portions of hexane and the rinses are added to the separating funnel. About 30 mL acetonitrile–saturated with hexane is added to it and the acetonitrile layer is drained into a 250 mL round bottomed flask containing

anhydrous sodium sulphate. The acetonitrile extract is evaporated to dryness at 60°C. The concentrated residue is dissolved in 5 mL hexane and cleaned up by adsorption column chromatography using 10 g of 5% deactivated florisil and 100 mL of 6% diethyl ether in hexane as eluting solvent. Prior to elution the column is washed with 50 mL of hexane to remove the co-extractives. The eluate collected is concentrated at about 60°C to dryness and diluted with 10 mL of hexane and injected into GLC, under the following conditions.

Instrument:

Perkin Elmer Clarus 500 GC

Detector

Electron Capture Detector (ECD)

Column

Elite-5(Cross bond 5% diphenyl - 95% dimethyl poly siloxane,30 m

length x 0.25 mm i.d $\times 1 \mu m$ film thickness)

Gas flow rate

Carrier (Nitrogen)

5 mL/min

Temperatures

Injector Detector 180°C 300°C

Oven :

210°C

Sample volume injected

 0.5μ L

3.2. Preparation of acetonitrile saturated with hexane

Three portions of acetonitrile combined with one portion of hexane in a 125 ml separating funnel. Gently shaken and then collect the lower part of acetonitrile layer. This is called acetonitrile saturated with hexane.

4. Calculation:

where,

As = "Sample" peak area corresponding to pesticide at its retention time

A std = "Standard" peak area corresponding to pesticide at its retention time

C= Concentration of pesticide in standard solution (ppm)

D = Sample dilution factor (mL)

W= Weight of tea sample (g) taken for analysis

In all the analysis, an equal volume of sample and standard solutions are injected into the GC.

SOP determination of imidacloprid residues in black tea and tea infusion (provided by India)

1. Introduction

This method is applicable for the determination of imidacloprid residue in black tea and tea infusion.

2. Principle

2.1. Black tea

Imidacloprid is extracted with acetonitrile from the black samples. The concentrated residue is determined by HPLC with DAD detector.

2.2. Tea brew

Two gram of made tea was infused in 100 mL of boiling water (ISO 3103 -1990). After 6 min of brewing, the water extract was filtered, cooled and partitioned with 100 mL of dichloromethane (twice). The organic phase was passed through anhydrous sodium sulphate. The extract was concentrated by evaporating in a rotary vacuum evaporator and diluted with 10 mL acetonitrile and analysed for the residues of imidacloprid. The spent leaves were dried between the folds of filter paper and residues were extracted following the method described above for black tea.

3. Extraction

Ten grams of tea sample is extracted with 100 mL of acetonitrile by shaking in a mechanical shaker for two hours. The contents are filtered, washed with 50 mL of acetonitrile and the extract is concentrated to dryness in a rotary vacuum evaporator with the water bath maintained at 85°C.

3.1. Clean up

The concentrated residue is dissolved in acetonitrile and transferred to glass column packed with activated florisil (10 g) for clean up. Top and bottom of florisil, 1 cm layer of anhydrous sodium sulphate is packed. The compound is eluted with 100 mL acetonitrile. After evaporation, the samples are suitable diluted with acetonitrile and analysed in HPLC.

Instrument

: HPLC (Agilent; Model 1100)

Detector

: Diode array detector

Column

: Zorbax Rx C18 (4.6 X 250mm)

Mobile phase: Acetonitrile: Water (35:65, v/v)

Flow rate

: 1.0 mL/min

Wavelength

: 270 nm

Injection volume $: 10 \mu L$

4. Calculation:

Concentration of

AsXCXD

residues (mg/kg) =

A std X W

where,

As = "Sample" peak area corresponding to pesticide at its retention time

A std = "Standard" peak area corresponding to pesticide at its retention time

C = Concentration of pesticide in standard solution (ppm)

D = Sample dilution factor (ml)

W= Weight of tea sample (g) taken for analysis

In all the analysis, an equal volume of sample and standard solutions are injected into the HPLC.

SOP for determination of dimethoate residues in black tea and tea infusion (provided by India)

1. Introduction

This method is applicable for the determination of dimethoate residue in black tea and tea infusion.

2. Principle

2.1. Black tea

Dimethoate is extracted with dichloromethane from the black tea samples. The extract is partitioned with hexane and cleaned up through silica gel column. Then it is eluted with dichloromethane. The concentrated residue in the eluate is determined by GC with nitrogen-phosphorus detector.

2.2. Tea brew

Two gram of made tea was infused in 100 mL of boiling water (ISO 3103 -1990). After 6 min of brewing, the water extract was filtered, cooled and partitioned with 100 mL of dichloromethane (twice). The organic phase was passed through anhydrous sodium sulphate. The extract was concentrated by evaporating in a rotary vacuum evaporator and diluted with 10 mL acetone and analysed for the residues of dimethoate. The spent leaves were dried between the folds of filter paper and residues were extracted following the method described above for black tea.

3. Extraction

Fifty grams of tea sample is extracted with 200 ml dichloromethane by shaking it in a mechanical shaker for two hours. The contents are filtered, to the filtrate add 1 g of activated charcoal, shake vigorously and the contents are filtered using Whatmann No. paper and washed with dichloromethane. The extract is passed through sodium sulphate funnel to a 500 ml round bottom flask. Add 3drops of propylene glycol and concentrate the extract to about 2 mL.

3.1. Clean up

To the concentrated residue in the round bottom flask, add about 50 mL of 15% aqueous methanol, shake vigorously. Transfer it to a 125 mL separatory funnel and add 40mL hexane and shake for 2 min. and allow the phases to separate. Drain the lower aqueous layer into a 500 mL round bottom flask. Repeat the process twice with an additional 40 mL of hexane;

drain the aqueous extract into the flask. Combine the aqueous extraction re-partitioned with 20mL of dichloromethane (thrice). Combine the dichloromethane extract and evaporate to near dryness in a rotary vacuum evaporator. The concentrated residue is dissolved in hexane and transferred to a glass column packed with activated silica gel (10 g) using dichloromethane. In between the silica gel, 1 cm layer of anhydrous sodium sulphate is packed. Prior to elution, the column is washed with 50 ml of dichloromethane to remove the co-extractives and the washings are discarded. The compound is eluted with 200 mL of dichloromethane. After evaporation, the samples are suitably diluted with acetone and analysed in GC as per the conditions given below:

Instrument

Hewlett Packard 5890 series II Gas

Chromatograph

Detector

Nitrogen Phosphorus Detector (NPD)

Column

HP-608 (PH ME Siloxane wide bore capillary

 $30 \text{ m} \times 0.53 \text{ mm} \times 0.5\mu$

:

Gas flow rate

Carrier (Nitrogen)

: 12.0 ml/min

Temperatures

Oven

180°C

Injector

225°C

Detector

200°C

Sample volume injected

 $0.5 \mu L$

4. Calculation:

Concentration of

As X C X D

residues (mg/kg) =

A std X W

Where,

As = "Sample" peak area corresponding to pesticide at its retention time

A std = "Standard" peak area corresponding to pesticide at its retention time

C = Concentration of pesticide in standard solution (ppm)

D= Sample dilution factor (ml)

W= Weight of tea sample (g) taken for analysis

In all the analysis, an equal volume of sample and standard solutions are injected into the GC.

Enclosure III

SOP for determination of bifenthrin residues in green tea and tea infusion (provided by China)

1. Introduction

This method is applicable for the determination of bifenthrin residue in green tea and tea infusion.

2. Principle

2.1. Green tea

Bifenthrin is extracted with acetone from the green tea samples. The extract is cleaned up through florisil column. Then it is eluted with hexane/acetone/toluene mixture. The concentrated residue in the eluate is determined by GC with electron capture detector.

2.2. Tea brew

Two gram of made tea was infused in 100 mL of boiling water (ISO 3103 -1990). After 6 min of brewing, the water extract was filtered, cooled, and transferred to the 250mL graduated cylinder, by adding 30g NaCl and 150mL acetonitrile, shake vigorously 1min, balance 30min, take upper 100mL of acetonitrile in 500mL round bottomed flask, The extract was concentrated by evaporating in a rotary vacuum evaporator with the water bath maintained at 45 °C, then N₂ drying. The spent leaves were dried between the folds of filter paper and residues were extracted following the method described above for green tea.

3. Extraction

Ten gram of green tea sample is extracted with 70mL of acetone by immersing overnight. The contents are filtered through anhydrous sodium sulphate layer to a 500 mL round bottomed flask. The extract is evaporated to dryness on a rotary vacuum evaporator and the residue is dissolved in 10 mL hexane.

3.1. Clean up

Prior to elution the column is washed with 10 mL of hexane to discard, then 1mL sample is transferred to 1g of 10% deactivated florisil column(1.0cm, $id \times 15$ cm). The hexane/acetone/toluene(445:10:45,V/V/V) mixture eluate collected in 10 mL flask, and injected into GC, under the following conditions.

Instrument: Varian CP3800 or other type of GC

Detector : Electron Capture Detector (ECD)

Column : DB-1701(30 m length x 0.32 mm i.d x 0.25 μ m

film thickness)

Gas flow rate

Carrier (Nitrogen) : 2 mL/min

Temperatures

Injector : 260°C Detector : 300°C

Oven : Initial temperature 80 °C, hold 1 min, at the 10 °C/ min

rate of temperature, raise to 150 °C, hold 1 min, then at the5 °C/

min rate of temperature, raise to 260 °C, hold 5 min;

Sample volume injected : 1µL

4. Calculation:

Concentration of residues (mg/kg)

$$Cx = \frac{Fa \times Cs \times Qs \times Vx \times Vex}{Fs \times Qx \times Vri \times M}$$

where,

Fa = "Sample" peak area corresponding to pesticide at its retention time

Fs = "Standard" peak area corresponding to pesticide at its retention time

Cs = Concentration of pesticide in standard solution (mg/L)

M= Weight of tea sample (g) taken for analysis

Qs: Standard Inject Volume, µL;

Vx: Sample Volume, mL;

Vex: Total Extraction Volume, mL;

Fs: Standard Area:

Qx: Sample Inject Volume, µL;

Vri: Part Extraction Volume, mL;

In all the analysis, an equal volume of sample and standard solutions are injected into the GC.

SOP determination of imidacloprid residues in green tea and tea infusion (Provided by China)

1. Introduction

This method is applicable for the determination of imidacloprid residue in green tea and tea infusion.

2. Principle

2.1. green tea

Imidacloprid is extracted with acetonitrile from the green tea samples by adding 20mL water. The extract is cleaned up through florisil column. Then it is eluted with hexane/acetone mixture. The concentrated residue in the eluate is determined by HPLC with DAD detector.

2.2. Tea brew

Two gram of made tea was infused in 100mL of boiling water (ISO 3103 -1990). After 6 min of brewing, the water extract was filtered, cooled and Transferred to the 250mL graduated cylinder, by adding 30g NaCl and 150mL acetonitrile, shake vigorously 1min, balance 30min, take upper 50mL of acetonitrile in 500mL round bottomed flask, The extract was concentrated by evaporating in a rotary vacuum evaporator with the water bath maintained at 40°C, then N₂ drying. The spent leaves were dried between the folds of filter paper and residues were extracted following the method described above for green tea.

3. Extraction

Weigh 5g tea sample in 150mL Centrifuge bottle, add 25mL water for soaking one hour, then add 50mL acetonitrile, homogenous with IKA-18 homogenizer for one minute, centrifuge for 5 minutes with 5000 rpm, the organic solvent transferred to the 100mL graduated cylinder and repeat to add 50mL acetonitrile and homogenate and centrifuge steps, add 20g NaCl to graduated cylinder, shake vigorously 1min, balance 30min, take upper 40mL of acetonitrile in 250mL round bottomed flask, The extract was concentrated by evaporating in a rotary vacuum evaporator with the water bath maintained at 45°C, N2 drying.

3.1. Clean up

The concentrated residue is dissolved in $3 \times 2mL$ hexane/acetone (4:1,V/V) and transferred to glass column packed with 5g of 11% deactivated florisil column(1.6cm, id×25cm) for clean up. On the top and bottom of florisil, 2 cm layer of anhydrous sodium sulphate is packed. The compound is eluted with 20 mL hexane/acetone (4:1,V/V) and discard, the elute with 50mL

hexane/acetone (1:1,V/V) was collected, and concentrated and evaporated with rotary vacuum evaporator with the water bath maintained at 40°C, the samples are diluted with 2mL acetonitrile and analysed with HPLC.

Instrument

: HPLC (Agilent 1100)

Detector

: Diode array detector

Column

: Aglient TC-C18 (4.6 X 250mm)

Flow rate

: 1.0 mL/min

Wavelength

: 270 nm

Mobile phase

: Gradient Elution Procedure::

Tooldie.:				
Setp	Total time	Flow rate (mL/min)	A: water (0.2% Phosphoric acid aqueous solution) %)	B: acetonitrile
0	0.00	1	90.0	10.0
1	30.00	1	40.0	60.0
2	35.00	1	90.0	10.0

Injection volume : 10 μL

4. Calculation:

Concentration of residues (mg/kg):

$$Cx = \frac{Fa \times Cs \times Qs \times Vx \times Vex}{Fs \times Qx \times Vri \times M}$$

where,

Fa = "Sample" peak area corresponding to pesticide at its retention time

Fs = "Standard" peak area corresponding to pesticide at its retention time

Cs = Concentration of pesticide in standard solution (mg/L)

M= Weight of tea sample (g) taken for analysis

Qs: Standard Inject Volume, µL;

Vx: Sample Volume, mL;

Vex: Total Extraction Volume, mL;

Fs: Standard Area:

Qx: Sample Inject Volume, µL;

Vri: Part Extraction Volume, mL;

In all the analysis, an equal volume of sample and standard solutions are injected into the HPLC.

SOP for determination of dimethoate residues in green tea and tea infusion (Provided by China)

1. Introduction

This method is applicable for the determination of dimethoate residue in green tea and tea infusion.

2. Principle

2.1. Green tea

Dimethoate is extracted with acetonitrile from the green tea samples by adding 20mL water. The extract is cleaned up through Envi-Carb SPE column. The concentrated residue in the eluate is determined by GC with flame phosphorus detector.

2.2. Tea brew

Two gram of made tea was infused in 100 mL of boiling water (ISO 3103-1990). After 6 min of brewing, the water extract was filtered, cooled and Transferred to the 250mL graduated cylinder, by adding 30g NaCl and 150mL acetonitrile, shake vigorously 1min, balance 30min, take upper 50mL of acetonitrile in 500mL round bottomed flask, The extract was concentrated by evaporating in a rotary vacuum evaporator with the water bath maintained at 40°C, then N₂ drying. The spent leaves were dried between the folds of filter paper and residues were extracted following the method described above for green tea.

3. Extraction

Weigh 5g tea sample in 150mL Centrifuge bottle, add 25mL water for soaking one hour, then add 50mL acetonitrile, homogenous with IKA-18 homogenizer for one minute, centrifuge for 5 minutes with 5000 rpm, the organic solvent transferred to the 100mL graduated cylinder and repeat to add 50mL acetonitrile and homogenate and centrifuge steps, by adding 20g NaCl, shake vigorously 1min, balance 30min, take upper 20mL of acetonitrile in 250mL round bottomed flask, The extract was concentrated by evaporating in a rotary vacuum evaporator with the water bath maintained at 45°C, N2 drying.

3.1. Clean up

The concentrated residue is dissolved in $3\times 2mL$ acetonitrile /hexane (3:1,V/V) and transferred to Envi-Carb SPE column for clean up. The compound is eluted with 25mL acetonitrile /hexane (3:1,V/V), the elute was concentrated and evaporated with rotary vacuum evaporator with the water bath

maintained at 45°C, the samples are diluted with acetonitrile and analysed in GC-FPD as per the conditions given below:

Instrument : Agilent 6890 N Gas chromatograph or other type of GC

Detector : Flame Phosphorus Detector (FPD) Column : HP-17 (30 m \times 0.32 mm \times 0.25 μ)

Gas flow rate

Carrier (Nitrogen) : 2.0 ml/min

Temperatures

Injector : 22°C

Detector : 250°C

Oven : Initial temperature 100 °C, hold 1 min. at the

30 °C/min rate of temperature, raise to 220 °C, hold 10 min, then at the 45 °C/min rate of temperature

raising, raise to 250 °C, hold 5 min:

Sample volume injected : 1.0µL

4. Calculation:

Concentration of residues (mg/kg)

$$Cx = \frac{Fa \times Cs \times Qs \times Vx \times Vex}{Fs \times Qx \times Vri \times M}$$

where.

Fa = "Sample" peak area corresponding to pesticide at its retention time

Fs = "Standard" peak area corresponding to pesticide at its retention time

Cs = Concentration of pesticide in standard solution (mg/L)

M= Weight of tea sample (g) taken for analysis

Qs: Standard Inject Volume, μL;

Vx: Sample Volume, mL;

Vex: Total Extraction Volume, mL;

Fs: Standard Area;

Qx: Sample Inject Volume, μL:

Vri: Part Extraction Volume, mL:

In all the analysis, an equal volume of sample and standard solutions are injected into the GC.