Progress of the validation of analytical chemical methods for testing and measuring cigarette contents and emissions

Report by WHO

1. At its third session (Durban, South Africa, 17–22 November 2008), the Conference of the Parties (COP) noted the information contained in the progress report\(^1\) of the working group on Articles 9 and 10 of the WHO Framework Convention on Tobacco Control (WHO FCTC), and decided\(^2\) to request the Convention Secretariat to invite WHO to validate, within five years, the analytical chemical methods for testing and measuring the cigarette contents and emissions identified as priorities in the progress report of the working group, using the two smoking regimens set out in paragraph 18 of that report, and to inform the COP through the Convention Secretariat on a regular basis of the progress made. There were three priorities identified with regard to cigarette contents (nicotine, ammonia and humectants) and five with regard to emissions in mainstream smoke of cigarettes (tobacco-specific nitrosamines (TSNAs), benzo[a]pyrene (B[a]P), aldehydes, volatile organic compounds (VOCs) and carbon monoxide).

2. Of the eight methods identified, the WHO Tobacco Laboratory Network (TobLabNet) validated the methods for carbon monoxide in 2007, TSNAs and nicotine in 2010,\(^3\) and B[a]P and humectants in 2012.\(^4\) The standard operating procedures (SOPs) for TSNAs and nicotine\(^5\) were completed in 2014. WHO is currently working on the validation of methods for ammonia in cigarette tobacco filler, and

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\(^1\) Document FCTC/COP/3/6.
\(^2\) See decision FCTC/COP3(9).
\(^3\) See the report by WHO’s Tobacco Free Initiative to the fourth session of the COP, document FCTC/COP/4/INF.DOC./2.
\(^4\) See the report by WHO’s Tobacco Free Initiative to the fifth session of the COP, document FCTC/COP/5/INF.DOC./1.
VOCs and aldehydes in mainstream cigarette smoke. This progress report contains the validation status of the three aforementioned methods.

3. After WHO has completed the validation work mandated by the COP for the methods, the SOPs and reports will be delivered shortly thereafter, depending on the availability of technical and financial resources. The publicly available final SOPs will be posted on the WHO and WHO FCTC websites.

Validation of a method for the determination of benzo[a]pyrene in mainstream cigarette smoke

4. The validation has been completed with the participation of eight laboratories, one each from Burkina Faso, Canada, China, France, Japan, Singapore, and two from the United States of America. The SOP is being finalized.

Validation of a method for the determination of humectants in tobacco

5. The validation has been completed with the participation of 13 laboratories for the Gas Chromatography Flame Ionisation Detection (GC-FID) method and seven laboratories for the Gas Chromatography Mass Spectrometry (GC-MS) method. The validation was successfully completed on two analytical instrumentation platforms, namely SOP 06: GC-FID, and SOP 06 bis: GC-MS. The humectants method validation was led by Burkina Faso and China, with the China National Tobacco Quality Supervision and Test Centre (CNTQSTC) performing the data processing and statistical analysis, with support from WHO and US Centers for Disease Control and Prevention. One of the humectants, triethylene glycol, was not detected in the reference cigarettes, so two cigarette samples with different levels of triethylene glycol were manufactured and supplied by China during the method validation process. The participating laboratories for GC-MS were from Burkina Faso, China, Greece, Japan, Singapore, and two from the United States. The participating laboratories for GC-FID were from Burkina Faso, Canada, two from China, France, Germany, Greece, Japan, Singapore, Spain, the Netherlands, and three from the United States. The laboratories from Burkina Faso, China, Greece, Singapore, and two laboratories from the United States participated in both validations. The SOP is currently being finalized.

Validation of a method for the determination of ammonia in tobacco

6. As a result of some technical challenges, the ammonia method validation is behind schedule. As reported before (in document FCTC/COP/5/INF.DOC./1), TobLabNet agreed to forego the enzymatic approach and start developing an ion chromatography technique for the ammonia validation. The validation was unable to proceed as planned in 2013 due to difficulties in finding laboratories with the necessary equipment to run the validation. Eventually, in February 2014, TobLabNet managed to gather eight laboratories to participate in this validation. The full validation started in June 2014 and the results are expected to be obtained in October 2014. The participating laboratories are from Canada, two from China, Greece, Indonesia, Japan, Spain, and the United States.

Validation of a method for the determination of volatile organic compounds and aldehydes in tobacco smoke

7. The National Institute of Public Health (NIPH) in Japan has invented a novel technique to trap both VOCs and aldehydes in cigarette smoke. This innovation consists of using one trapping material – Carboxen 572 ® which is a type of treated carbon.1 Adaptation was needed to apply this novel technique to rotary and linear smoking machines. Colleagues from CNTQSTC and Laboratoire

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of National Essais (LNE) in France adapted this new technique by re-designing the trap to enable it to be used in smoking machines. China supported the preparation of this device and distributed it to participating laboratories to carry out this validation work. Extra time was needed for research and development on the pad/cartridge holder and as a result of the logistics involved in the manufacture and assessment of each pad/cartridge holder. The adaptation was made possible with good cooperation and significant efforts by LNE in France, CNTQSTC in China, NIPH in Japan, and the National Institute of Public Health in the Netherlands. With the ammonia validation now in progress, validation of VOCs and aldehydes will resume after completion of the ammonia validation. The participating laboratories are from Burkina Faso, Canada, two from China, France, Japan, Singapore, the Netherlands, and the United States.

8. At the seventh meeting of the WHO Study Group on Tobacco Product Regulation in December 2013 in Rio de Janeiro, Brazil, it was recommended that among methods that do not exist yet, priority should be given to the development by TobLabNet of standardized testing methods for the measurement of:

   (a) cadmium and lead content in tobacco;

   (b) nicotine in smoke of waterpipe (shisha); and

   (c) nicotine, TSNAs, and B[a]P in smokeless tobacco products.¹

**Action by the Conference of the Parties**

9. The COP is invited to note this report.

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¹ See document FCTC/COP/6/14.