International Journal of Science and Qualitative Analysis
2018; 4(3): 93-99
http://www.sciencepublishinggroup.com/j/ijsqa
doi: 10.11648/j.ijsqa.20180403.15
ISSN: 2469-8156 (Print); ISSN: 2469-8164 (Online)

Inter-Laboratory Study for the Asbestos Detection in Bulk Materials: First Italian Scheme

Antonella Campopiano[1, *], Angelo Olori[1], Biagio Maria Bruni[2], Luciano Benini[3], Mauro Campanella[4], Fulvio Cavariani[5], Adriano Fava[6], Patrizia Garofani[7], Luigi Leone[8], Claudio Martinelli[9], Mariano Alessi[10]

[1]Department of Medicine, Epidemiology, Occupational and Environmental Hygiene, National Institute for Insurance against Accidents at Work (INAIL), Monte Porzio Catone, Rome, Italy

[2]Department of Technology and Health, National Health Institute (ISS), Rome, Italy

[3]Asbestos Regional Center of Marche, ARPAM Pesaro, Italy

[4]Asbestos Regional Center of Abruzzo, ARTA Teramo, Italy

[5]Asbestos Regional Center of Lazio, ASL Viterbo, Italy

[6]Asbestos Regional Center of Emilia Romagna, ARPA Reggio Emilia, Italy

[7]Asbestos Regional Center of Umbria, AUSL 2 Assisi, Italy

[8]Asbestos Regional Center of Basilicata, ARPAB Potenza, Italy

[9]Asbestos Regional Center of Veneto, ARPA Verona, Italy

[10]General Direction of Health Prevention, Italian Ministry of Health, Rome, Italy

Email address:

a.campopiano@inail.it (A. Campopiano), a.olori@inail.it (A. Olori), biagio.bruni@iss.it (B. M. Bruni), luciano.benini@ambiente.marche.it (L. Benini), maurocamp@alice.it (M. Campanella), labig@asl.vt.it (F. Cavariani), afava@arpae.it (A. Fava), patrizia.garofani@uslumbria1.it (P. Garofani), luigi.leone@arpab.it (L. Leone), cmartinelli@arpav.it (C. Martinelli), alessi@sanita.it (M. Alessi)

*Corresponding author

To cite this article:

Antonella Campopiano, Angelo Olori, Biagio Maria Bruni, Luciano Benini, Mauro Campanella, Fulvio Cavariani, Adriano Fava, Patrizia Garofani, Luigi Leone, Claudio Martinelli, Mariano Alessi. Inter-Laboratory Study for the Asbestos Detection in Bulk Materials: First Italian Scheme. *International Journal of Science and Qualitative Analysis*. Vol. 4, No. 3, 2018, pp. 93-99. doi: 10.11648/j.ijsqa.20180403.15

Received: May 2, 2018; **Accepted**: May 29, 2018; **Published**: June 19, 2018

Abstract: Italian Health Ministry established by regulation the minimum requirements for laboratories intending to perform asbestos analysis and the organization of a quality control scheme to which the laboratories must refer. Three proficiency testing schemes for asbestos detection in bulk materials using polarized light microscopy (PLM), X-ray diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) were developed according to the regulation of Health Ministry. The schemes were organized in rounds. In each round the laboratory received one batch of 4 samples. The participant laboratories were 475. The performance of laboratory as satisfactory, unsatisfactory or awaiting classification. The samples of the rounds were real world materials from abatement project and might contain any of the asbestos types. Ceiling-tile containing vitreous fibers caused the highest frequency of false positive; 87% of errors incurred in FTIR and 60% in XRD. Vinyl-floor tiles caused most false negative; 100% of errors incurred in PLM in the second pilot program, 13% in FTIR and 10% in XRD. One of the main factors causing variability of results turned out to be related to the skill of the analysts. Training and experience of the analysts help to get a reliable and reproducible asbestos analysis.

Keywords: Asbestos Containing Materials, Quality Control Scheme, PLM, XRD, FTIR

1. Introduction

Until the 1980s, asbestos, because of its high technical performances, was used extensively, in Italy, for building and industrial applications. A major worldwide use of asbestos in manufacturing was in combination with cement to form a variety of asbestos-cement products, such as pipes, flat and corrugated sheets. Other significant uses of asbestos were in asphalt and vinyl-based flooring, in insulating products for pipes and boilers, in roofing felts, in special textiles, in friction materials, in spray composition fireproofing products and in electrical insulations.

Exposure to asbestos fibers may result in a diffuse interstitial fibrosis of the lung parenchyma (asbestosis), fibrosis of the pleura (pleural plaques or diffuse pleural thickening) and cancer (mesothelioma and bronchogenic carcinoma). These pathologies were already known in the fifties and sixties and are well described in numerous publications [1-6].

The International Agency for research on Cancer (IARC) classified all forms of asbestos carcinogenic to humans (Group 1) [7].

To assess the extent and severity of the health risk, the asbestos in materials is detected and airborne asbestos fibers concentration is measured.

Surveys must be carried out by competent personnel namely must have sufficient training, qualifications, knowledge, experience as described in Health and Safety Executive guideline [8].

Proficiency testing programs [9-11] are considered essential to assuring the quality of the analysis, especially for data related to health public decisions.

The Italian Ministry of Health established by regulation the minimum quality requirements for laboratories intending to perform asbestos analysis and general organization of the quality control scheme to which the laboratories must refer [12].

The minimum quality requirements relate to instrumentation and staff laboratory. Each laboratory must be equipped with the right instrumentation and fulfill minimum quality requirements. The Ministry of Health recognized the analytical techniques to be used for asbestos determination in bulk and airborne samples: the phase contrast optical microscopy (PCM), Polarized Light Microscopy (PLM), the scanning electron microscopy (SEM); the X-ray diffraction (XRD); the Fourier Transform Infrared Spectroscopy (FTIR).

The laboratories that provide airborne asbestos analysis and qualitative asbestos analysis in bulk samples must be equipped with a PCM or/and a SEM equipped with an X-ray microanalysis system.

The laboratories that provide asbestos quantitative analysis in bulk samples must be equipped with a XRD or/and FTIR. Moreover the laboratories must have the auxiliary apparatuses for the performance of the tests and measurements required for bulk asbestos analysis.

The laboratory staff have to consist of a graduated in technical or scientific disciplines and a coworker with an upper school leaving certificate, both of them with specific and evident proof experience in the techniques of optical microscopy, and/or electron microscopy, and/or X-ray diffraction, and/or infrared spectroscopy.

The first national quality control was done by two pilot projects coordinated by National Institute for Insurance against Accidents at Work (INAIL), Department of Occupational and Environmental Medicine, Epidemiology and Hygiene.

This paper wants show the analytical performance of bulk asbestos laboratories and identify some common problems that arise during asbestos containing material analysis.

2. Methods

The pilot scheme for asbestos analysis in bulk material was prepared by INAIL, the National Health Institute (ISS), the Ministry of Health and the Asbestos Regional Centres (ARC) of each Region, identified as regional centers specialized in the risk assessment of human asbestos exposure. The participant laboratories to two pilot projects were 475.

The scheme had the following phases: 1) registration of the participating laboratories in the program; 2) on-site assessment of laboratory; 3) preparation and choice of the samples; 4) dispatch of the samples to participants by the ARC; 5) sample analysis by the participating laboratories; 6) gathering of the participant laboratories resulting by the ARC; 7) processing of the results; 8) evaluation of the performances of the participating laboratories. The scheme was organized in rounds. A round consisted of 4 different samples.

The performance of laboratory was satisfactory, unsatisfactory or awaiting classification by comparing laboratory analysis with reference analysis. The awaiting classification provided a further round.

The purpose of the on-site assessment was to determine if the laboratory was following its documented management system and to assess the technique competence of the staff members according to national decree [12].

The on-site assessment was carried out by ACR and took account of the management structure of the laboratory, the standard operating procedures including the safe work and emergency procedures, the worker training and education, the equipment for bulk sampling analysis and for air sampling analysis.

The samples of the rounds were among these in Table 1. Over 2000 samples were prepared. The samples were real world materials from abatement project and might contain any of the asbestos types. The materials for the most part were removed from buildings and were representative of the samples that a typical laboratory encounters.

The samples used in this scheme contained amounts of asbestos, when it was present, at levels above 5%.

All samples are fully analyzed and validated by a team of experienced analysts of the reference laboratories (INAIL, ISS and ACR) using more than one analytical technique.

The results of the analysis carried out from the reference laboratories were compared and if no significant difference existed, the material was used for the test.

Participant laboratories had approximately 7 working days to analyze the samples and to report their results.

Participants were required to report: proper classification of the sample as asbestos-containing or not asbestos-containing, proper identification of asbestos types present, if any, and finally quantification of asbestos present. The latter request was optional and few laboratories gave the result.

No specific test method but specific analytical technique was required for the analysis of the samples. The samples should be analyzed using the same procedure used for their routine samples.

Table 1. *Bulk materials used in two pilot schemes for asbestos analysis.*

Material	Target component
fiber-cement	no asbestos
asbestos-cement	chrysotile
asbestos-cement	chrysotile and crocidolite
vinyl floor tile	chrysotile
vinyl floor tile	no asbestos
plaster	amosite and synthetic vitreous fibers
ceiling tile	no asbestos (synthetic vitreous fibers)
spray-on insulation	amosite
coating	no asbestos (aramidic fibers)
reinforced resin	amosite and crocidolite

3. Results

The Figure 1 and 2 show the participant laboratories to the two pilot projects by Region; 255 to the first and 220 to the second respectively.

158 laboratories identified asbestos in bulk materials using PLM, 39 laboratories using XRD and 86 using FTIR in the first pilot project (Figure 3); and 94 (PLM), 21 (XRD) and 109 (FTIR) in the second pilot project (Figure 4).

The performance of the laboratories was "satisfactory" if the laboratory made no analytical error. The types of analytical errors were qualitative errors (false negative and false positive), and asbestos identification errors. False positive result means to report asbestos when none is present; the contrary happens for false negative result.

The failure to identify the asbestos component in a sample containing asbestos generated the rating of "awaiting classification", for FTIR and XRD program, with a further round. For PLM program, "awaiting classification" was given to the laboratories that made any analytical error on a sample. In all other cases the laboratory was "unsatisfactory".

The unsatisfactory laboratories of the first pilot program were 21% for FTIR program, 3% for PLM program and no unsatisfactory laboratory for XRD program.

In the second pilot study the percentage of the unsatisfactory laboratories were 35% using FTIR, 33% using PLM and 40% using XRD. Qualitative and identification errors were frequent.

The analysis of asbestos is a complex task which involves different approaches depending on the characteristics of the bulk samples. Bulk samples with low asbestos content are the most problematic. The samples selected for these quality assurance programs contained amounts of asbestos, if asbestos were present, at levels above 5%.

Results from each pilot program were examined and the highest frequencies of false positive, false negative and asbestos identification errors were identified. The samples causing the highest frequencies of false positive using FTIR were ceiling tile containing vitreous fibers (87% of errors incurred in FTIR program), the percentage decreased when the analysis was in XRD (60% of errors) in the second pilot program.

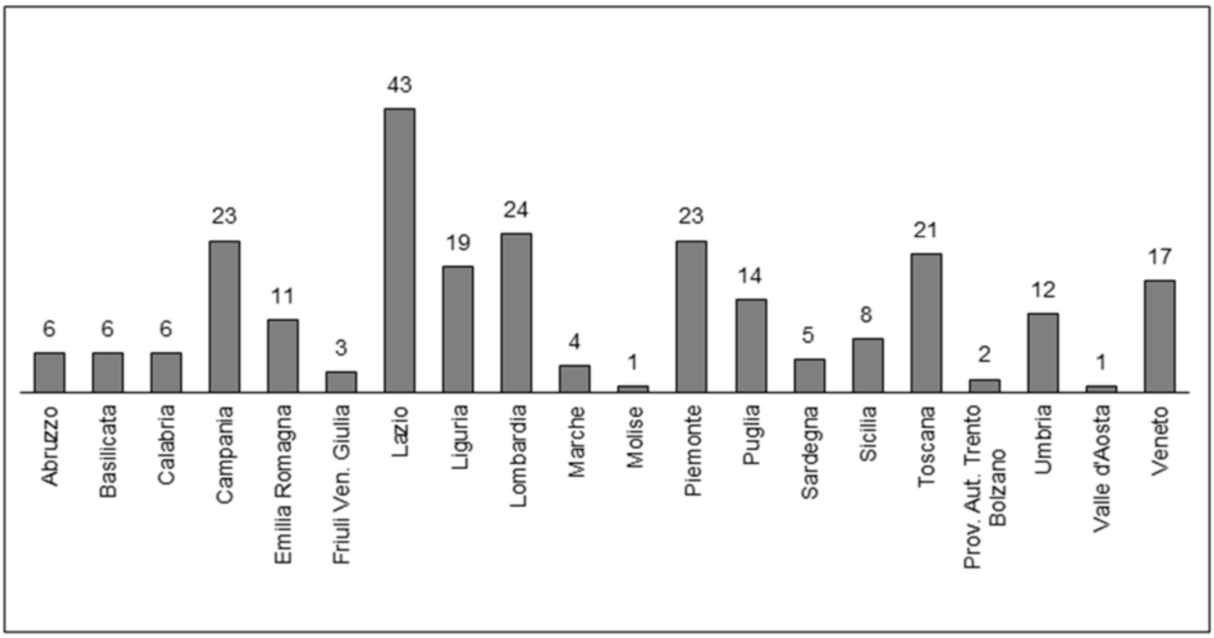

Figure 1. *Number of participant laboratories by Italian Region in the first pilot project.*

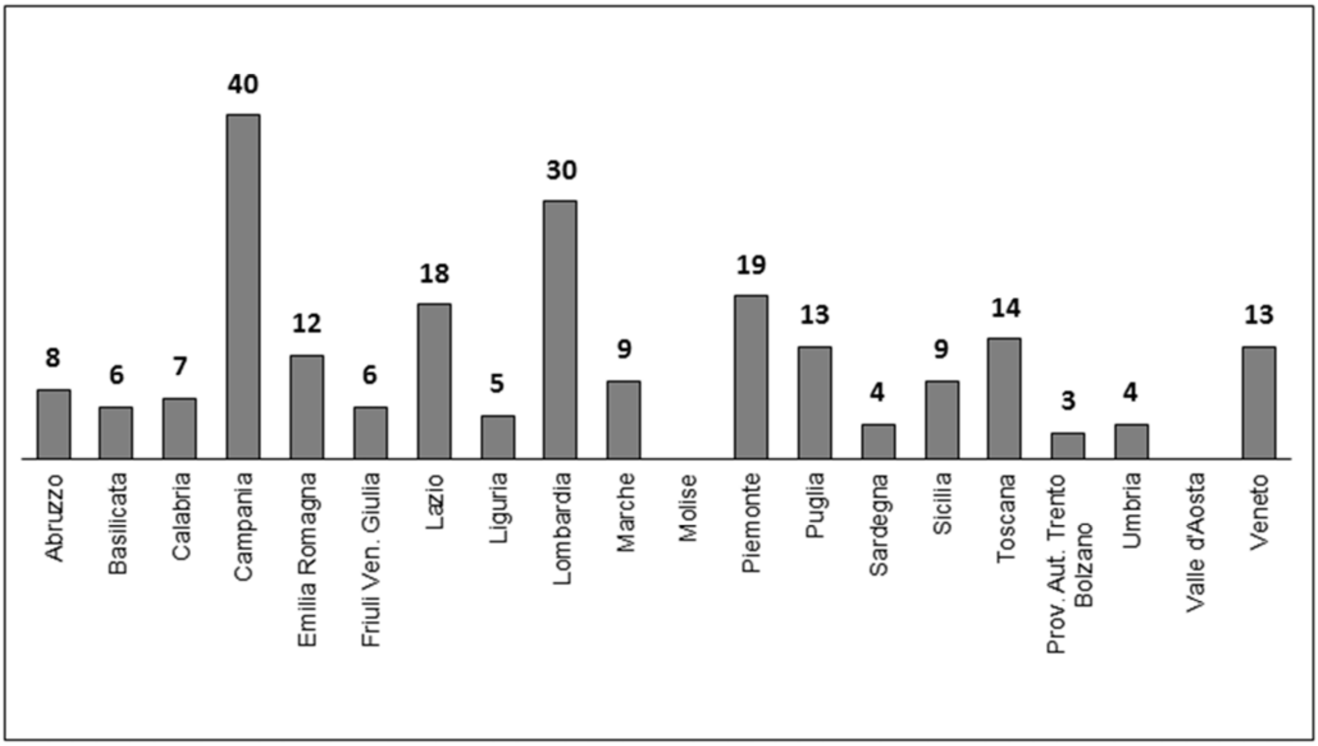

Figure 2. *Number of participant laboratories by Italian Region in the second pilot project.*

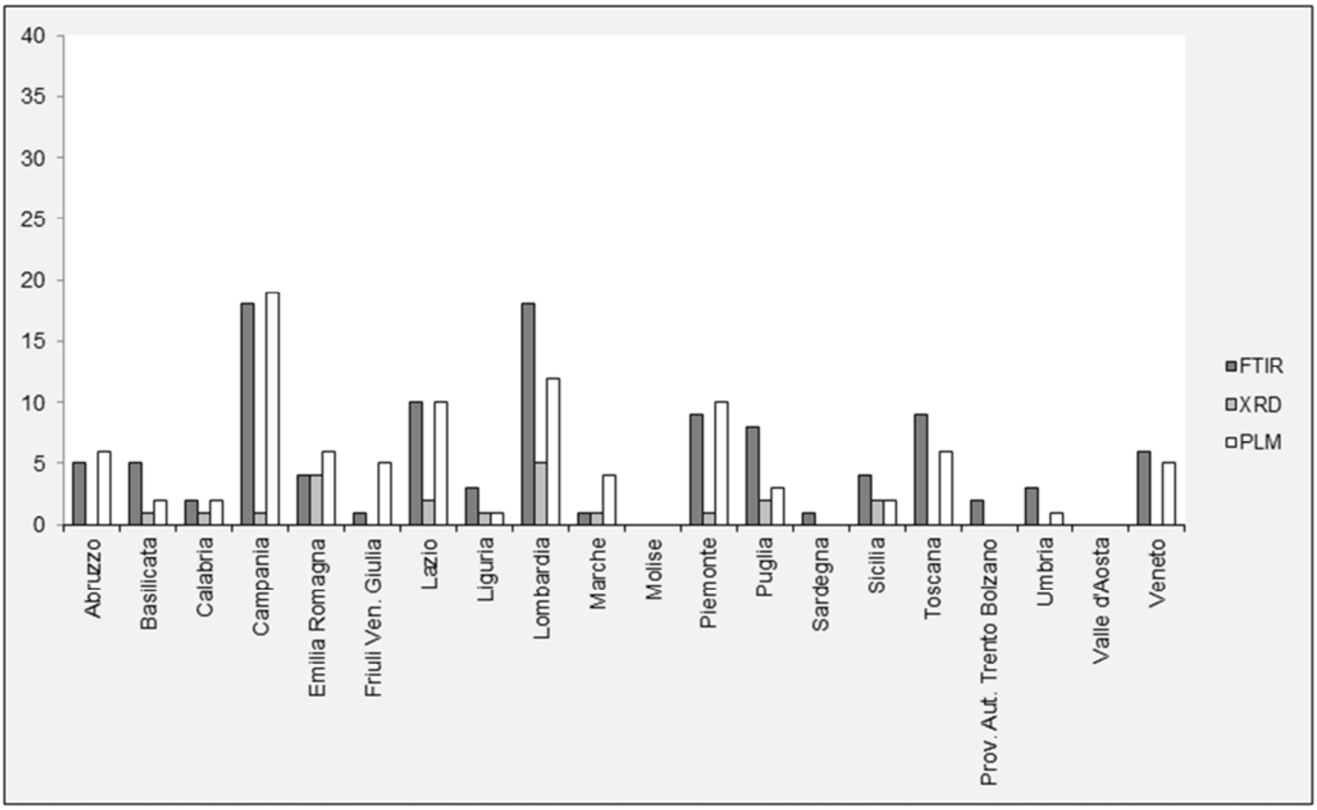

Figure 3. *Number of participant laboratories in FTIR, XRD and PLM schemes by Italian Region in the first pilot project.*

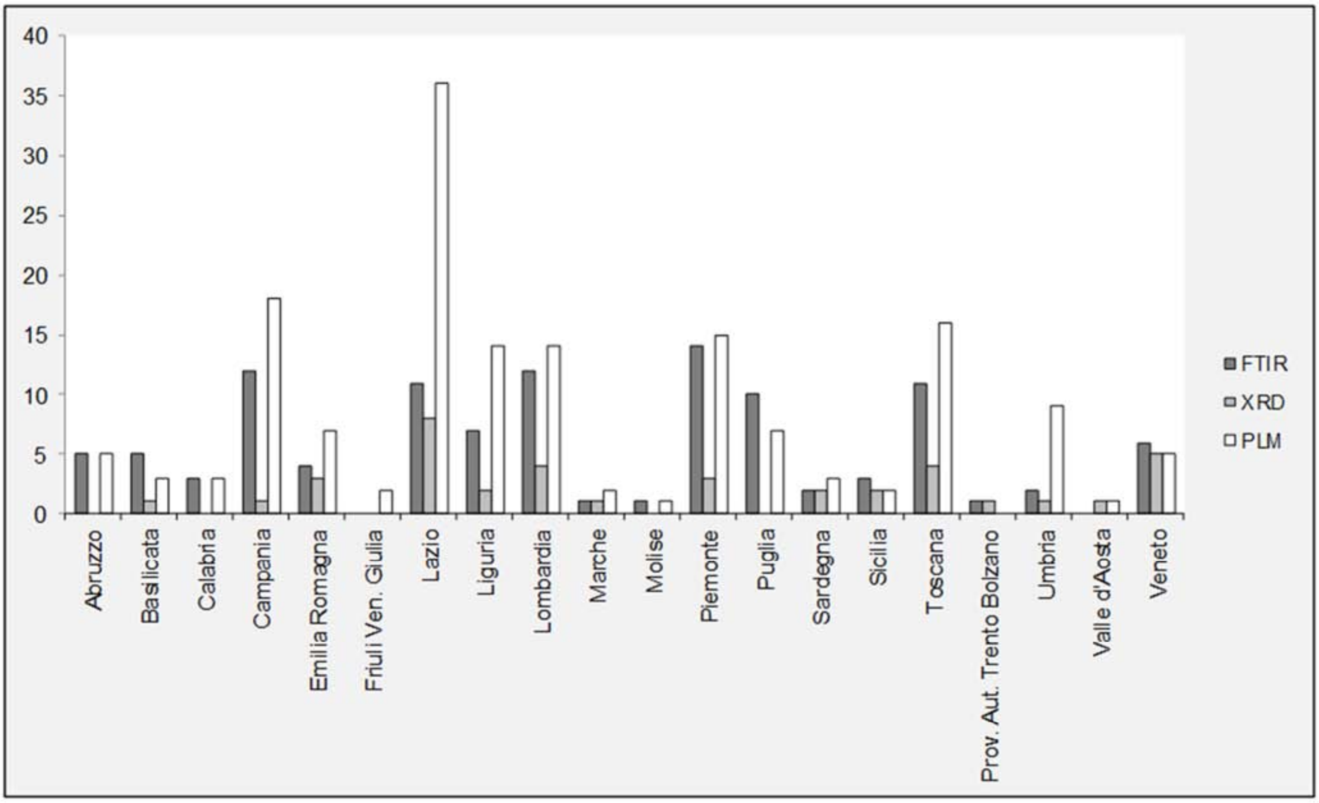

Figure 4. *Number of participant laboratories in FTIR, XRD and PLM schemes by Italian Region in the second pilot project.*

The reason of the analytical errors occurred in FTIR might be attributed to the presence in the sample of some interferences such as kaolinite or similar mineral types [13]. Kaolinite is widely used in several industrial products and it is plausible to find this mineral within ceiling tile. In all spectra of kaolinite, there are four typical vibrational regions of the mineral. The region of OH stretching (3700–3620 cm^{-1}), SiO stretching (1120–1000 cm^{-1}), OH bending (940–910 cm^{-1}) and SiO_2 bending (550–400 cm^{-1}). The false positive arises from misinterpretation of the peaks in the first region, near to the OH stretching of chrysotile. An expert analyst studies the spectrum fingerprint of the sample where the typical peak of chrysotile at 606 cm^{-1} assigned to inner Mg–OH vibration, is absent.

The type of asbestos-containing material that caused most false negative was vinyl floor tiles. In the second pilot program these materials were responsible for 100% of errors incurred in PLM program; 13% of errors incurred in FTIR and 10% of errors incurred in XRD.

Vinyl matrix of these materials tenaciously binds to the fibers making very difficult detection of asbestos. The asbestos present in these materials is always chrysotile and the percentage is typically low. The asbestos fibers are often short end thin. The whole of these factors hinder asbestos detection by PLM.

Generally, optical microscopy is the main method to detect the presence of asbestos in bulk building material. Polarized light allow the microscopist to distinguish asbestos from non-asbestos fibers using the appropriate Cargille refractive index liquid [14, 15]. With this method, a small amount of the sample is crushed and placed on a microscope slide, but for the vinyl tiles it is better to scrape transversely the sample with a lime in order to better liberate the fibers from the matrix.

It must be kept in mind that asbestos analysis is not a simple chemical analysis but it is an analytical process. The general flow of the analysis is:

Gross examination;

1. Examination under polarized light on the stereo microscope;
2. Examination by phase-polar illumination on the compound phase microscope;
3. Determination of species by dispersion stain;
4. Difficult samples may need to be analyzed by scanning or transmission electron microscopy, or the results from those techniques combined with light microscopy for a definitive identification.

The Figure 5 shows the suggestion of Crane [16].

The identification of a particle as asbestos requires that it be asbestiform. XRD and FTIR are applicable techniques for quantitative measurement or auxiliary to microscopy but they cannot give the information about size or shape (fiber or not). The coupling of more analytical techniques is the key for a good asbestos analysis.

4. Conclusions

The Laboratories were called upon to identified asbestos in a variety of bulk materials. The analysis of asbestos is a complex task which involves different approaches depending on the characteristics of the bulk samples. One of the main factors causing variability of results turned out to be related to the skill of the analysts. The experience of the analyst is essential. Unfortunately, many analysts working on bulk samples lack a scientific attitude and a scientific background. Few of them are mineralogists, some or no chemical background. These inexpert operators have considerable difficulty unless they develop the proper scientific approach with proper training.

The participating laboratories in the first pilot program had an evident proof experience in the analytical techniques. The first pilot program, carried out in 2008, collected the laboratories operating in Italy since the 80s and 90s; while the second pilot program in 2011 collected new laboratories less skillful. Training and experience of the analysts help to get a reliable and reproducible asbestos analysis.

Accredited laboratories, and the specific methods the accreditation covers, are listed at www.salute.gov.it of the Ministry of Health.

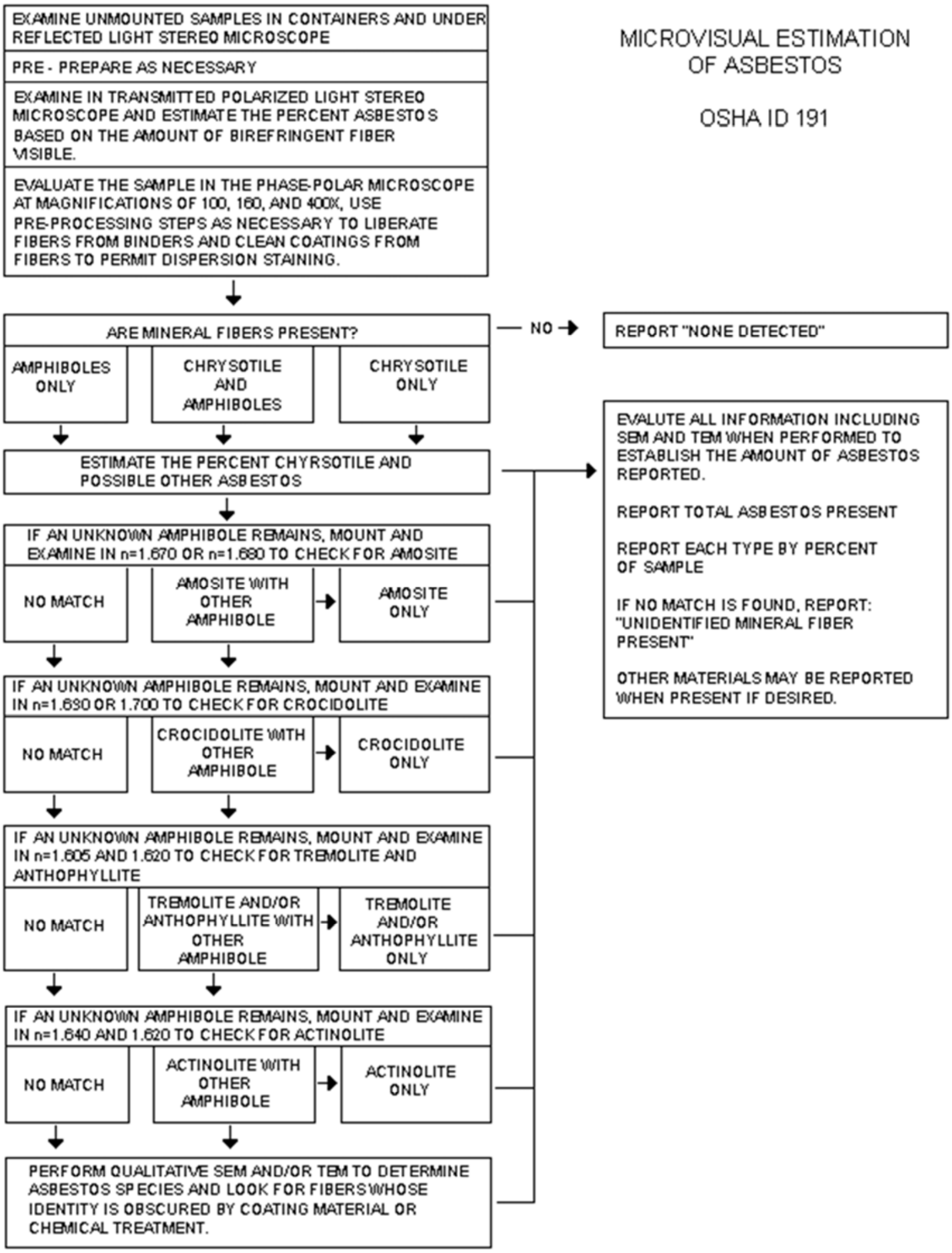

Figure 5. *General flow of the asbestos analysis suggested from Crane. From OSHA Method number ID 191 [16].*

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