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AZEOTROPIC DISTILLATION FOR THE MEASUREMENT OF FREE WATER IN CHRYSOTILE ASBESTOS

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SUMMARY OF RESULTS

Because of the difficulties encountered in determining moisture in asbestos, another method was investigated. This method, azeotropic distillation, appears to have good potential for moisture determination. It is simple, at least as rapid as the oven drying method and has less inherent error.

Round robin testing should be done to validate the conclusions reached in this study.

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INTRODUCTION

The moisture content of asbestos is extremely important to both its usage and sales. Because of the special nature of asbestos, the exact determination of moisture is difficult and inconsistent results are common. Chrysotile absorbs and desorbs moisture readily; moisture content is therefore dependent on atmospheric conditions. Since this product is tested and shipped world-wide, this parameter is very important. Because the current technique for moisture determination is not completely satisfactory, a different method (used in the coal industry) was examined for its potential application in the asbestos industry.

HISTORICAL

A testing procedure utilizing a drying oven has been standardized and approved by the Canadian Asbestos Industry[1] for moisture determination in asbestos fibre. A quantity of asbestos is dried for one hour at 105°C to 110°C, dessicated, and reweighed. The moisture content is then calculated. Recently a moisture method essentially similar to the above has been examined by ASTM[2]. In this method, atmospheric temperature and humidity conditions are defined so that results from different parts of the world can be compared.

The azeotropic distillation method, the subject of this report, has been used in a different discipline[3]. This technique was modified for asbestos fibres.

SAMPLES

The two samples of fibre used for this study were selected at random from a group of Quebec asbestos samples. These were samples A and B, both from the vicinity of Thetford Mines, Quebec.

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(1) Testing Procedures for Chrysotile Asbestos Fibre; by A.T.I., Min. Fib. Prod. Bureau, and QAMA (1966). Available from QAMA, Quebec City.
(2) ASTM - Committee D30 (Sub. 3).
(3) "Distillation with Toluene" - Britain Standards, 1016, Part I (1957).

EXPERIMENTAL

The Dean-Stark apparatus, shown in Figure 1, was used. A 500-ml round-bottomed flask fits into an electric mantle-heater which is controlled by a variable transformer. The Dean-Stark distillation and receiving tube, which is inserted into the flask, can be seen. This standard apparatus is listed in any laboratory equipment catalogue.

Initially, 5 g of asbestos fibre was put into a flask containing 200 ml of toluene. The mixture was refluxed for one hr and then allowed to stand for 2 hr. The amount of sample and liquid and the refluxing time were varied to obtain optimum conditions. Glass beads were used to prevent bumping during the boiling period. The oven drying method for determination of moisture content was used as a control.

RESULTS AND DISCUSSION

The observations and results of the experiments are shown in Tables 1, 2, and 3.

Table 1 shows the amount of moisture present in the two samples using the oven drying method.

TABLE 1

Moisture Determination of Asbestos
Oven Drying (Control) Method

Sample	Wt Used g	Loss in Weight g	Average Moisture %
A	1	0.0065	0.67
"	1	0.0069	
B	1	0.0061	0.60
"	1	0.0059	
A	25	0.2000	0.80
B	25	0.2000	0.80

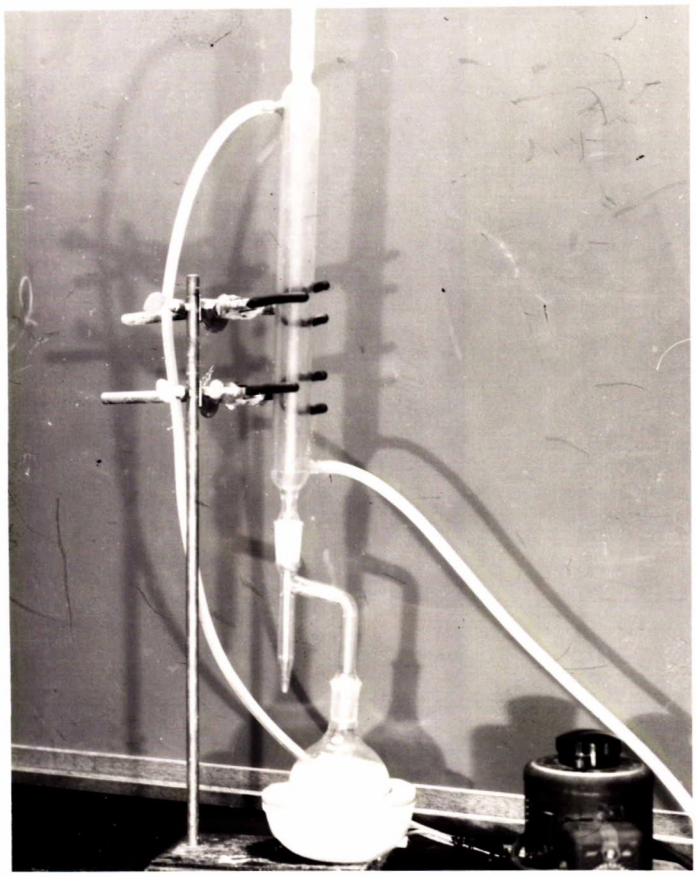

Figure 1. Dean Stark Apparatus for Azeotropic Distillation of Chrysotile Asbestos.

Table 2 summarizes the results of the initial distillation experiments.

TABLE 2

Azeotropic Distillation

Sample	Liquid and Boiling Point	Amount Liquid ml	Wt Fibre g	Distillation Period min	Water Collected ml	Remarks
A	Toluene BP 110°C	200	5	60	less than 0.1	No reading possible;
A		200	5	180		Fibres dark green after distillation;
B	Xylene BP 141°C	300	25	180	0.3	Fibres light brown after distillation;
A		300	25	180		Fibres light green after distillation;

After the above initial tests, more detailed experiments were performed on new samples. Twenty-five g of asbestos and 300 ml of liquid toluene and xylene (sufficient to cover the sample) were used. The results are summarized in Table 3.

TABLE 3

Summary of Results

Distilling Time min	Amount of Water Distilled in Column (sample wt 25 g)							
	Xylene				Toluene			
	B		A		B		A	
	ml	%	ml	%	ml	%	ml	%
0	-	-	-	-	-	-	-	-
5	0.05	0.2	0.05	0.2	0	0	0	0
15	0.10	0.4	0.10	0.4	0.10	0.4	0.10	0.4
30	0.10	0.4	0.10	0.4	0.15	0.6	0.15	0.6
45	0.15	0.6	0.15	0.6	0.20	0.8	0.20	0.8
60	0.20	0.8	0.15	0.6	0.20	0.8	0.20	0.8
75	0.25	1.0	0.20	0.8	0.25	1.0	0.25	1.0
90	0.30	1.2	0.20	0.8	0.25	1.0	0.25	1.0
120	0.30	1.2	0.20	0.8	0.25	1.0	0.25	1.0
150	0.30	1.2	0.20	0.8	-	-	-	-
180	0.30	1.2	0.20	0.8	-	-	-	-
after standing 30 min	0.30	1.2	0.30	1.2	0.30	1.2	0.30	1.2

Except in a few instances, xylene mixtures resulted in more water being distilled. This is to be expected because the boiling point of xylene is greater than that of toluene.

Upon completion of the distillation period and after standing for 30 min, a small additional amount of water was noted in the water column. In every case the final water reading was the same (0.30 ml). The amount of water distilled reached equilibrium, after 75 min, with toluene.

The asbestos samples had obviously been dehydrated during storage and contained very little free water. This is indicative of the type of problem to be expected with asbestos fibres. The moisture content of asbestos fibre normally ranges from 10 to 15 per cent.

The oven drying method has a number of steps where possible errors may arise. The first possible error, that in initial sample weighing, is common to both methods. However, the oven drying method stipulates that drying should always be done at one temperature and for a definite period of time. The sample must then be dessicated and reweighed. All these steps can produce errors that result in some difficulty in interlaboratory comparisons. In the azeotropic distillation technique, however, the mixture of liquids boils at one temperature. No dessication and no further weighing are required and the amount of water distilled can be read directly from the graduated column. Atmospheric conditions therefore have very little bearing on this method. The toluene can also be separated from the water very easily and reused, if desired.

Azeotropic distillation appears to result in slightly more water than the oven drying method. However, it cannot be said that the oven drying method is more accurate or more consistent. Another control method, e.g., the Karl Fischer, the partial pressure technique, or the reaction of water with calcium carbide, can be used independently to indicate the true amount of free moisture.

The wet, coloured samples of asbestos, after distillation, were put into an oven dryer at 110°C and allowed to dry for two days. All samples were fully dry at the end of this period and all had reverted to their original predistillation colour. It would seem that the liquids were either adsorbed on the surface or that a reaction had occurred between the asbestos and liquid; perhaps, both adsorption and reaction could take place.

To learn more about this interesting phenomenon, the dried samples were observed under ultra-violet light. The results are summarized in Table 4.

TABLE 4

Fluorescence of Asbestos (Toluene and Xylene Mixtures) after Drying and Observed under Ultra-Violet Light

Ultra-Violet Light	Samples	
	B	A
Long wave - Toluene	slightly fluorescent	no fluorescence
Long wave - Xylene	" "	slight "
Short wave - Toluene	more than with long wave	slight "
Short wave - Xylene	most fluorescent of all samples	more fluorescent than above

Although no definite explanation can be made at this time, it is suspected that fluorescence of the dried samples is due to the reaction of the xylene with asbestos and that, even after drying, at least part of the reaction product did not revert to its original form.

SUMMARY

The conclusions reached are tentative only because the asbestos samples appear to have been partially dehydrated in storage.

Toluene, which boils at about 110°C, appears to be slightly better than xylene for the purpose of moisture determination in asbestos. Further testing should be done to confirm this. The distillation method resulted in more free water from the asbestos than did the oven drying method but it is uncertain which method is superior; another control method might establish this.

There is less error inherent in the distillation technique and the results obtained with this technique appear to be consistent. The distillation does not take much longer (75 min) than the oven drying time (60 min) and does not require any further steps such as dessication and reweighing. Therefore, the total time for the azeotropic distillation technique for moisture determination should be less than for oven drying.

The liquids used (toluene, xylene) seem to react with asbestos, in some cases, to change the color of the asbestos to a light brown or green. Drying of the sample reverses this color change. The fluorescence in some of the samples is probably due to a reaction between the liquid and the fibre.

Azeotropic distillation appears to have good potential as an alternate method for the determination of free moisture in chrysotile asbestos. However, "round robin" testing should be done to confirm this.

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