

ETHYL CORPORATION

Inter-Office

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Dr. R. A. Kehoe ← THIS COPY FOR
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To: Mr. H. K. Ball
From: R. L. Hudson
Subject: Lead in Air Analysis

New York
Baton Rouge
July 27, 1960

We were pleased to receive your letter of July 22. It appears that we have a good number of problems before we have the solid iodine LIA method in the field, but I'm sure we can work this out. What is of immediate concern to us is that the standard old LIA method is still being used in California for TML in air, and I quote from your letter: "Therefore, there would be no difference at all in the method used for taking TEL in air and the recommended method for this survey of taking TML in air. At J. Cholak's suggestion our people were to take 8 cu. ft. reading every 10th sample." You further state that the above information is based on a letter received from A. H. Chenault, June 28, 1960.

Perhaps it would be of interest to you, and other parties concerned with this problem, to review the facts as to the standard LIA method for TML in air versus the modified tentative method which we have developed and recommended to be used until such time as the solid iodine scrubber method is available.

As early as February 6, 1959, we reported in the weekly letter to G. F. Kirby, and I quote: "Laboratory tests of the standard field kit method for lead in air have shown a serious error when applied to the measurement of TML in air. The efficiency of recovery of known amounts of TML vapor is only 10 to 25% under standard test conditions". The above data was later confirmed at our Detroit laboratories and is still essentially correct today.

On February 18, 1959, we reported in a teletype to Dr. Kehoe essentially these same results, stating that the results would be less than 25% of the true value, further pointing out that the reaction of TML with iodine is a slower reaction, and that several variables in the field kit are more critical for TML --- such as sampling rate, iodine concentration, glass wool packing, and temperature.

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On February 25, 1959, Dr. M. E. Griffing of our Detroit laboratories passed the above information to Dr. M. R. Barusch of the California Research Corporation, and further recommended to him:

- a. Use the scrubbers and solution recommended in the lead in air kit.
- b. Use two scrubbers in series and analyze their solutions separately. Consider any analysis suspect if the second scrubber contains more lead than the first.
- c. Use a fine glass wool; make sure that adequate packing is used, and that channelling does not occur.
- d. Use a flow rate of 0.3 cu. ft. per minute for low concentrations of TML and of 0.1 cu. ft. or less for high concentrations.

The above essentially describes what we consider to be a modified tentative method for TML in air, and we have adequate data to prove that recoveries with this system are in the 90% range. We have also considered other systems, such as 2 normal iodine and 1.5 normal KI in one scrubber. Also, we considered the use of alcoholic iodine. Both of these approaches were later abandoned, primarily because the solutions are difficult to handle and not readily available in the field except as bulk reagents.

On April 7 and 8 on your visit to Baton Rouge, you were told that the present LIA kit for TML in air is not adequate and that we would have to use some modifications, such as two scrubbers in series and low sampling rates. It was at this time that we also informed you that we could collect and measure TML quantitatively with the use of a solid iodine scrubber. Later this same month we provided Wil Hancock with the tentative field kit method for TML in air using two scrubbers in series, standard reagents, and a sampling rate of not more than 0.3 cu. ft. per minute, and a sample size of two cubic feet.

In May of this year we also supplied A. H. Chenault the same method along with enough bulk reagents for 400 TML in air tests.

Now to review..... We have always maintained that the present LIA method is inadequate for the measurement of TML in air and gives recoveries of less than 25% when used at standard (0.5 to 1.0 cu. ft./min.) sampling rates. We have recommended for an interim period a method using two scrubbers in series and low sampling rates, and have adequate data to prove that this tentative method gives approximately 90% recovery of TML in air. We have a method using solid iodine scrubbers which will give quantitative recovery of TML in air.

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The problem here, as you know, is to find a supplier who can manufacture these solid iodine scrubbers so that the scrubbers will not have an excessive pressure drop.

Finally, the new LIA method has been written and submitted to the Journal of Analytical Chemistry, and will shortly appear in public print. The article contains data that conclusively proves that our present LIA method is no good for TML, as well as a number of other organolead compounds in air.

R. L. Hudson

RLH/meb

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