AN ATOMIC ABSORPTION METHOD FOR THE DETERMINATION OF LEAD IN AIR

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Air pollution is increasing because of world-wide urbanization and continually increasing industrialization. The quantitative and qualitative composition of the contaminants present in the air depends on the degree and type of industrialization. The air of great cities contains Cu, Fe, Pb, Mn, Zn in high percentages, while Ba, Bi, Cd, Cr, Ni, Sn, Ti and V in smaller amounts. [1]

From among the elements mentioned, especially Bi and Pb are dangerous for the human organism. Therefore the knowledge of their amount in the air is of great importance. In recent years several papers have been published on the determination of lead as a contaminant of air.

Pines and Schwanecke [2] [3] used colorimetric methods for the determination of lead in air. Blumer [4] suggested a complexometric method, Jungreis and West [5] a ring-oven method, while Sciaraffa and Ziegler [6] developed an X-ray fluorescence method.

Atomic absorption methods have been developed by several authors for the same purpose. Japanese researchers, Ishii and Musha [7] injected the air sample containing lead directly into the flame of the atomic absorption apparatus. The atomic absorption methods developed by Den Tonkelaar et al. [8], further by Della Fiorentina [9] and Burnham et al. [10] as well as by Kaffenke [11] differ mainly in the way of sampling.

Flameless atomic absorption excitation was applied by Woodriff and Lech [12] as well as by Loftin and Christian [13]. Purdue et al. [14] measured the Pb content of air samples after enrichment by extraction.

We also developed an atomic absorption method for the determination of lead in air, and the data from sampling at a busy spot of Budapest are published below.

Experimental

Reagents

1. Strong nitric acid solution of analytical grade
2. Strong hydrogene peroxide solution

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3. 0,5% tartaric acid prepared with ion- exchanged water

4. Calibration solutions: a 1000 mg Pb/litre stock solution was prepared from lead nitrate of analytical grade, of which calibration solution series 0, 2.5, 5.0, 7.5, 12.5 and 15.0 mg Pb/l were diluted containing 0.5% tartaric acid.

Sampling

The air has been sampled by means of a sampling device developed by FEHÉR et al. [15] at the Central Research Institute for Physics.

The sampling device is a small pump driven by a motor, accumulator-fed. An air sample of 0.5—1 m^3 was pumped through a membrane filter type Synpor-3 into the head of the sampling device. The sampling head is connected to the pump through a flexible plastic tube. The sampling device was carried by persons on street duty at one of the busiest spots of Budapest.

The Central Research Institute for Physics provided the membrane filter sheets for analytical purposes.

Apparatus

The determinations were made at the resonance line 217.0 nm of lead by means of an atomic absorption device type UNICAM SP 90 A. The optimum parameters of the apparatus were established in preliminary experiments, the flow rate of the air was 5 litre/min, that of acetylene gas 1,8 litre/ min. The heating current of the hollow cathode lamp was 6 mA, the measurements were carried out in a height of 6 mm above the burner top.

To facilitate readings a five-fold scale extension was applied.

Process of preparation and measuring

The membrane filters were destructed in the 3 : 2 mixture of strong nitric acid and hydrogen peroxide in a Kjeldahl flask over a micro flame. After destruction, the remaining acid was carefully distilled, and the residue was washed with ion- exchanged water into small Petri- dishes, then evaporated to dryness under an infrared lamp. The dry residue was then filled up to 2.0 ml by 0.5% tartaric acid and the lead content of the samples was determined by atomic absorption method.

The evaluation was performed by means of a calibration set containing 0.5% tartaric acid.

Results and discussion

Effect of tartaric acid on the sensitivity of the determination

According to our experience, the sensitivity of lead determination by atomic absorption method little increases in the presence of tartaric acid, as seen from the comparison of calibration curves in Fig. 1.

Curves I and II indicate the absorption values of the calibration series without and with 0.5% tartaric acid respectively. The increasing sensitivity may be attributed to the formation of tartarate complexes. To test this fact, the absorption of 5×10^{-2} mmol/litre Pb-solutions was measured in tartaric acid of changing mol ratios. The sensitivity of the determination is increasing until formation of a Pb-tartarate complex of 1 : 1 mol ratio. Further addition of tartaric acid did not improve the sensitivity of the determination.

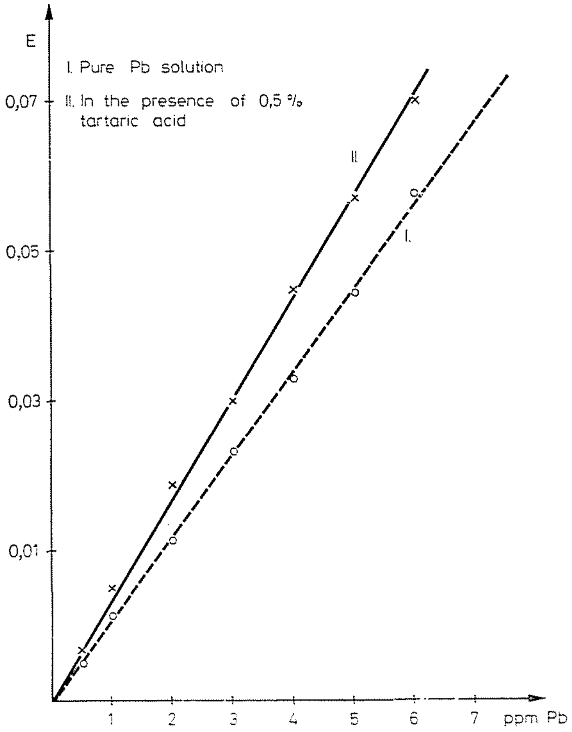


Fig. 1

The increase of the determination sensitivity was found to be about 13%. (Fig. 2)

Measurement results

The examined samples contained 0.6 to 13 μg of lead depending on the amount and on the pollution of air driven through. In a tartaric acid solution 0,65 μg of lead causes 1% absorption.

The reproducibility of the method has also been determined. Known amounts of lead were brought by a micropipette upon 9 membrane filter sheets

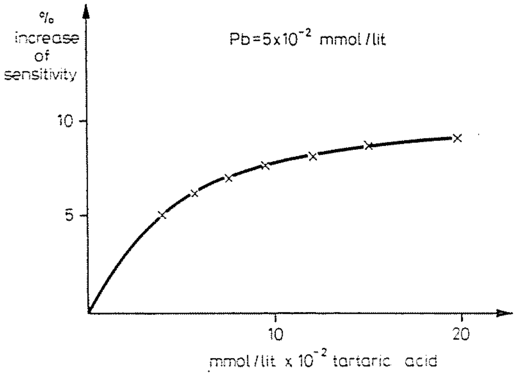


Fig. 2

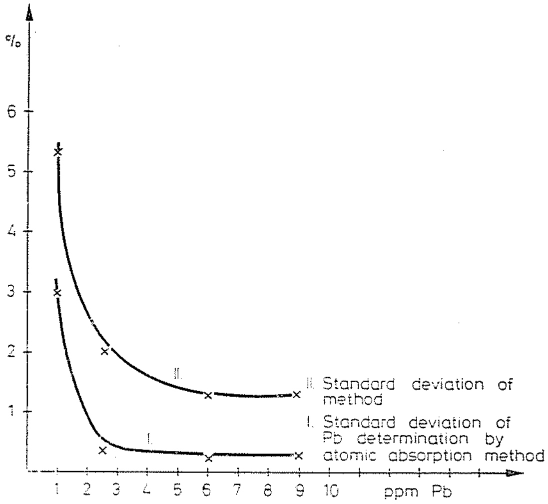


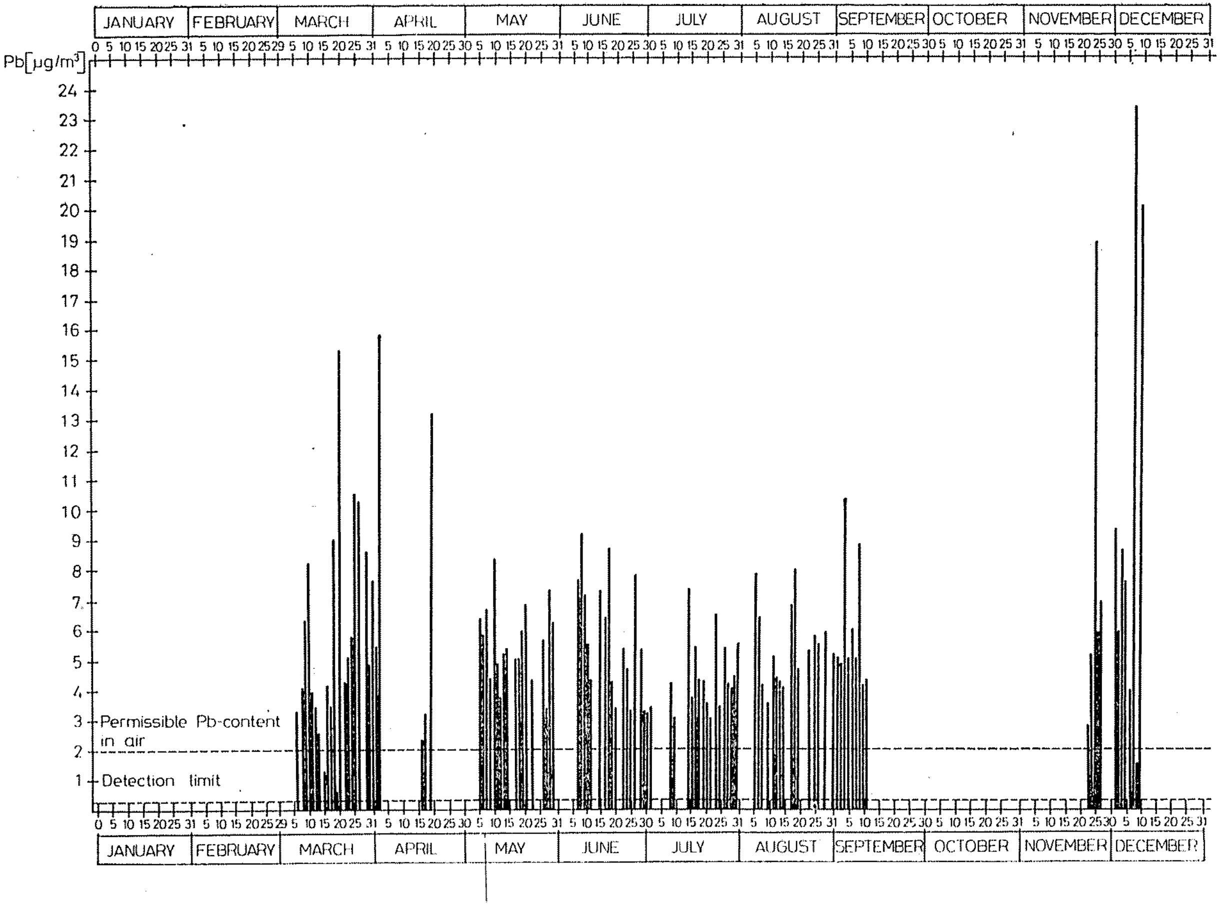
Fig. 3

each, and after destruction their lead content was determined. The reproducibility of the method is shown in Fig. 3.

The standard deviation of solutions concentrated at 1 to 3 μg Pb/ml was in the 3 to 5% range. The preparation by destruction increased the error of the determination by about 1%. The detection limit was at 0.5 μg/ml Pb content. From the about 180 air sample analyses the histogram of the lead concentration of air at a certain spot in Budapest downtown for 1971 has been constructed, shown in Fig. 4.

The lead contamination is given in Fig. 5. averaged for seasons.

In summer months the values were lower in spite of the heavier traffic. This phenomenon may be attributed to the upward air flow stronger in summer than in winter months.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Pb[μg/m³]
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Permissible Pb-content in air
Detection limit

Fig. 4

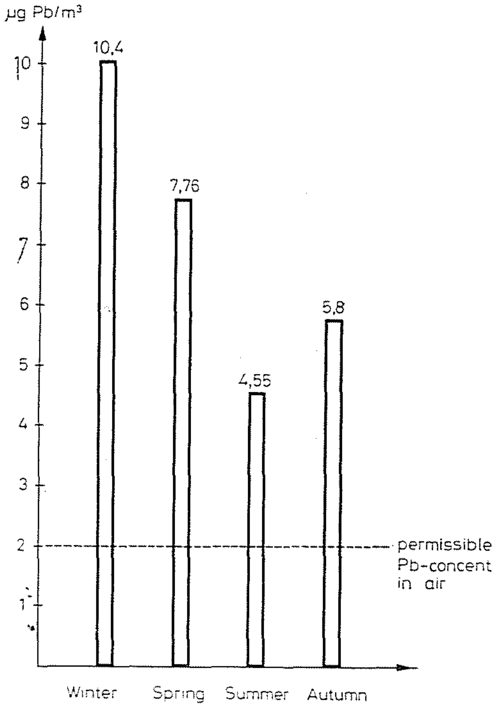

Fig. 5

Summary

An atomic absorption method has been elaborated for the determination of lead contamination in air. The air sample is pumped through a membrane filter type Synpor-3. The filter is destroyed in the 3 : 2 mixture of strong nitric acid-hydrogen-peroxide, the acid distilled and then the residue dried and filled up to 2 ml with 0.5% tartaric acid solution.

The results are evaluated by means of calibration curves. This method gives a standard deviation of 3 to 5% in 1 to 3 µg/ml concentration: in 0.5% tartaric acid solution the sensitivity is 0.65 µg Pb/ml 1% absorption. The presence of tartarate increases the sensitivity of the determination by 10 to 13%.

A histogram has been constructed of the lead contamination in air at one of the busiest spots of Budapest in 1971.

The lead content of the air was found to be lower in summer, attributed probably to the intenser air currents.

References

1. Meisel, T.—Pungor E., MTA Biol. Oszt. Közl. **14**, 63 (1971).
2. Pines, I.: Med. Pracy, **19**, (3) 229 (1968).
3. Schwanecke, R.: Arbeitsschutz, No. **7**, 169 (1969).
4. Blumer, W.: Präventiv-Medizin **14** (5) 303 (1969).
5. Jungreis, E.—West, P. W.: Isr. J. Chem. 7 [3] 413 (1969).
6. Sciaraffa, P. L.—Ziegler, C. A.: Isotop. Radiat. Technol. **8(2)**, 163 (1970).
7. Ishii, T.—Musha, S.: Bunseki Kogaku **19**, (10), 1436 (1970).
8. Den Tonkelaar, W. A. M.—Bikker, M. A.: Atmos. Environm. **5**, (5), 353 (1971).
9. Della Fiorentina, H.: Trib. CEBEDEAU (Cent. Belge Etude Doc. Eaux) **23**, (324) 482, (1970).

10. Burnham, C. D.—Moore, C. E.—Kowalski, T.—Krasniewski, J.: Appl. Spectroscopy **24**, (4) 411 (1970).
11. Kaffanke, K.—Nietruch, F.: Staub- Reinhaltung der Luft, **53**, (1), 33 (1973).
12. Woodriff, R.—Lech, J. F.: Anal. Chem. **44**, (7) 1323 (1972).
13. Loftin, H. P. Jr.—Christian, C. M.—Robinson, J. W.: Spectrosc. Letters, **3**, (7) 161 (1970).
14. Purdue, L. J.—Enrinoe, L. R., Thompson, R. J., Bonfield, B. A.: Anal. Chem. **45**, (3) 527 (1973).
15. Mészáros, I.—Biró, J.—Fehér I.: Nukleáris Gépészeti Konf. 1970. nov. 17—19. Budapest

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