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# COMBUSTION AND IR DETECTION METHOD FOR TOTAL ORGANIC CARBON DETERMINATION IN VARIOUS ENVIRONMENTAL MATRICES

Georgiana Cernica, Agnes Serbanescu, Adriana Cuciureanu, Bogdan Stanescu, Gina Catrina

National Research and Development Institute for Industrial Ecology-ECOIND 57–73 Drumul Podu Dambovitei Street, district 6, 060652, Bucharest, agnes.serbanescu@incdecoind.ro, Romania

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## *Introduction*

A fast and high performance method was developed for the determination of total organic carbon from different matrices (sludge, soil, treated bio-waste, filter ash). Total organic carbon is a key environmental indicator for soil, sludge and sediment and a parameter for characterizing sludge for energy recovery.

## *Materials and methods*

The developed method was based on the removal of inorganic carbon forms with a diluted non-oxidizing mineral acid (generally 10% $H_3PO_4$) before carbon content measurement. The principle of carbon measurement method is based on samples combustion and the analysis of the combustion gases, using infrared absorption. During combustion, the carbon components present in the sample are oxidized to form $CO_2$. The combustion temperature was 1350°C. The total carbon dioxide released during combustion was expressed as a percentage of carbon.

## *Results and conclusions*

The analyser was calibrated for a hight calibration range using calibration standard like: calcium carbonate, sulfanilic acid, EDTA and BBOT, and for a low calibration range using soil calibration standard with different carbon content. The calibration standard weight was around 100 mg. The performance parameters for the calibration curves, like intercept, slope, correlation coefficient are presented in Table 1 and Figure 1 for the high concentration range curve, and in Table 2 and Figure 2 for the low concentration range curve. A very good linearity was obtained for the two calibration curves, determination coefficients R2 was higher than 0.999.

**Table 1.** Performance parameters for high concentration range method

| Performance parameters | Value |
|---|---|
| Measuring range | 12-73% |
| Linear regression equation | Y=2.8387x-0.9236 |
| R correlation coefficient | 0.9999 |
| Residual standard deviation, $S_Y$ | 0.7929 |
| Standard deviation, $S_{x0}$ | 0.2795 |
| Coefficient of Variation, RSD | 0.7456% |

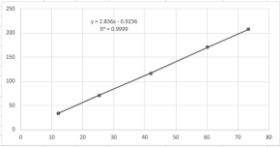

**Fig.1.** Calibration curve

**Table 2.** Performance parameters for low concentration range method

| Performance parameters | Value |
|---|---|
| Measuring range | 0.83-15.95% |
| Linear regression equation | Y=20.454x-1.1153 |
| R correlation coefficient | 0.9999 |
| Detection limit, LOD | 0.10 mg |
| Quantitaion limit, LOQ | 0.30 mg |
| Residual standard deviation, $S_Y$ | 1.053 |
| Standard deviation, $S_{x0}$ | 0,0515 |
| Coefficient of Variation, RSD | 0.61% |

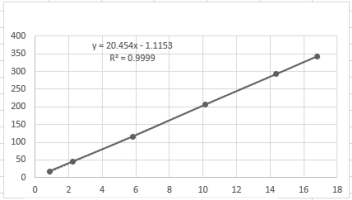


**Fig. 2.** Calibration curve

The samples were air-dried and milled using the Retsch Vibratory Disc Mill RS 200 in order to obtain a laboratory sample with a grain size of less than 200 μm.
The results and the performances for total organic carbon content determination in analysed samples (municipal solid sludge, soil, treated bio-waste and filter ash) are presented in Table 3.

**Table 3**. Performance parameters determined for total organic carbon content in analyzed samples

| Sample | $X_{mean}$ % | $S_r$ % | r % | $C_{Vr}$ % | $S_R$ % | R % | $C_{VR}$ % |
|---|---|---|---|---|---|---|---|
| Sludge | 23.02 | 0.32 | 0.89 | 1.38 | 0.38 | 1.06 | 1.65 |
| Treated biowaste | 47.65 | 0.47 | 1.32 | 0.99 | 0.55 | 1.54 | 1.16 |
| Soil | 2.46 | 0.13 | 0.37 | 5.35 | 0.19 | 0.52 | 8.18 |
| Filter ash | 0.35 | 0.011 | 0.031 | 3.22 | 0.016 | 0.044 | 4.42 |

The long-term precisions are very similar to the short-term precisions for all the analyzed sample. The developed method has many advantages such as the possibility of using a large amount of sample which makes it possible to analyze less homogeneous sample, short time of analyze, use of oxygen as a carrier gas, very good performances.

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