

# Online-Analysis at its best

## Online TOC Measurement in the Food Industry

The monitoring of Total Organic Carbon (TOC) in effluent streams offers many advantages over the traditional techniques of Chemical and Biological Demand (COD and BOD respectively). Not least is the possibility of obtaining analytical results within as little as five minutes compared with 5 days for a true BOD test or 2 hours for a COD determination. The use of online TOC analysers allows the effluent treatment process to be closely monitored and action can be taken to prevent discharges which would exceed the consent limit or disrupt the balance of later stages of the process.

The TOC-4100 Total Organic Carbon Analyser has been employed in a variety of industrial treatment plants including those dealing with waste water from municipal waste, chemical, pharmaceutical, paper and food manufacturing plants. Many of these applications are difficult in terms of the sample handling requirements as the effluent streams contain high levels of particulate matter which create severe challenges in terms of system design to ensure reproducible and representative measurements.

The TOC-4100 instrument has overcome many such challenges and an example of an application in the meat processing industry is described below.

### Theory of TOC Measurement

TOC is a direct measurement of the amount of organic carbon in a

sample and is therefore an important indicator of the organic pollution in water samples. A typical water sample will contain organic carbon compounds e.g. pollution in plant effluent and inorganic carbon compounds i.e. carbon dioxide and carbonates which occur naturally. The inorganic carbon content is not usually of interest.

**There are two principal methods for measuring TOC:**

#### 1. TC - IC (Difference Method)

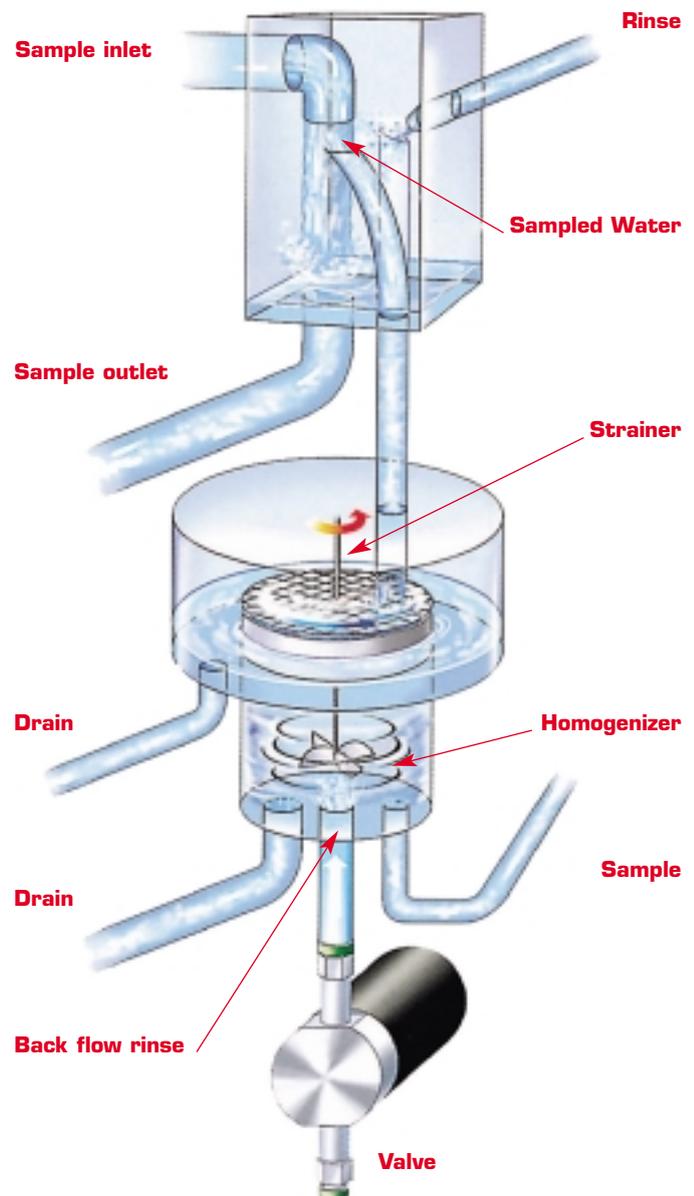
Where  $TOC = TC - IC$

TC = Total Carbon i.e. organic & inorganic carbon  
IC = Inorganic Carbon i.e. carbon dioxide & carbonates

This method involves two sequential measurements, TC followed by IC, and TOC is calculated.

The Shimadzu TOC-4100 employs the well established method of oxidation at 680° C using a platinum catalyst. Carbon compounds are injected onto the catalyst and converted to carbon dioxide which is measured using a non-dispersive infrared detector. The combination of the temperature and catalyst ensures a very high recovery rate even for difficult to oxidise compounds with high molecular weights.

Inorganic carbon is measured by adding hydrochloric acid to the sample in the syringe and sparging with air. The resulting carbon dioxide is measured by the infra-red detector.



Structure of a suspended particle flow line switcher with homogeniser

## 2. NPOC + POC (Addition Method)

Where  $TOC = NPOC + POC$

NPOC = Non-Purgeable Organic Carbon  
POC = Purgeable Organic Carbon

This method again involves two stages but the POC content in most cases is negligible so  $NPOC = TOC$ . The NPOC method is usually the quickest and most frequently used.

The measurement of NPOC is performed by acidifying the sample with hydrochloric acid and sparging with air to remove the inorganic carbon. The remaining non-volatile or non-purgeable carbon compounds are injected onto the catalyst and the carbon dioxide measured.

If there are volatile organic carbon compounds present these will be removed by the above process and will not be detected. It is possible using the POC option to pass these over a catalyst and measure them as they are being removed.

### Measurement of TOC in a Food Processing Factory

The application involves controlling the treatment of primary effluent from a meat processing factory. Raw effluent is pumped into a DAF tank and aerated while polymer is added to coagulate organic material which is skimmed from the surface and collected as a sludge.

The polymer was pumped at a constant rate and the plant opera-

tor wanted to improve control of the process by having the capability of varying the addition rate according to the organic loading in the tank. This will be beneficial for the secondary stages of the treatment process as it will prevent over or underfeeding of the micro-organisms. The operating regime involved taking manual samples on a daily basis and measuring the COD using a laboratory instrument. The test takes 2 hours to complete and due to the short residence time in the tank of 45 minutes it is not possible to control the process sufficiently. Also, given the nature of the plant there will be large fluctuations in the organic content of the effluent depending upon the time of day and stage of the process.

The sample stream is very variable in composition containing changing levels of fats, cooking oils, cleaning reagents and sewage. The sample is initially passed through a 1mm screen which can result in the formation of long filaments. These factors presented very severe challenges in sampling for any online analyser and several changes to the standard instrument were required.

Initially the standard TOC-4000 analyser was used with "suspended particle option". This system had proved itself in the chemical, pharmaceutical, paper, food and municipal waste industries among others.

The suspended particle option includes a high speed rotating mesh filter which is backflushed between samples and homogenising unit which breaks down particulates prior to introduction into

the analyser. The standard unit operates with a by-pass flow of 10 litres per minute and periodically the sampling tube moves into the flow to allow sample into the homogeniser unit.

However, it was found that with the standard arrangement it was occasionally possible for a filament to become attached to the top of the tube preventing further sample entering the homogeniser. A modification was employed which had proved successful in the paper industry where large "fines" are encountered with similar problems occurring as a result. The sampling system was modified to increase the sampling tubes from 6mm to 18 mm internal diameter and the inlets and outlets of the homogeniser were also increased in size. Pneumatically operated diaphragm valves with a large internal diameter were installed for stream switching. This solved the problem of the filaments and the sample was now reliably introduced into the homogeniser.

However, new problems were encountered inside the homogeniser due to the nature of the sample. The high levels of fats were causing gradual blocking of the filter and cold water washing was found to be insufficient for reliable operation. Two further modifications were installed which included washing with hot water and in addition to this a third stream containing 7 % HCl is introduced every 6 hours. This is treated as a normal sample and is taken through the syringe and injected onto the catalyst resulting in reactivation of the platinum sites. The frequency of the stream

switching is freely programmable so the cleaning interval can be increased or decreased accordingly.

The sample is acidified and sparged and analysed for non-purgeable organic carbon. The measurement takes approximately five minutes and the inlet and outlet streams are switched automatically. The results are printed out and also sent as 4-20 mA signals to a PLC where the trends are displayed. The instrument is programmed to calibrate automatically and fault alarms are available for connection to the control room.

A correlation between the manual COD measurements and the online TOC results has been found with a factor of approximately  $COD = 2.6 \times TOC$  and work is continuing to implement control of the dosing pump.

### Summary

The Shimadzu TOC-4100 analyser has been proved to operate reliably under difficult operating conditions measuring sample streams containing high levels of particulates and fatty materials.

The combination of a reliable sampling system and high temperature oxidation ensures that reproducible TOC measurements are obtained and a working correlation with laboratory COD results has been established.

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