

ON-LINE ANALYSIS IN THE PHARMACEUTICAL, FINE CHEMICAL AND FRAGRANCE INDUSTRIES

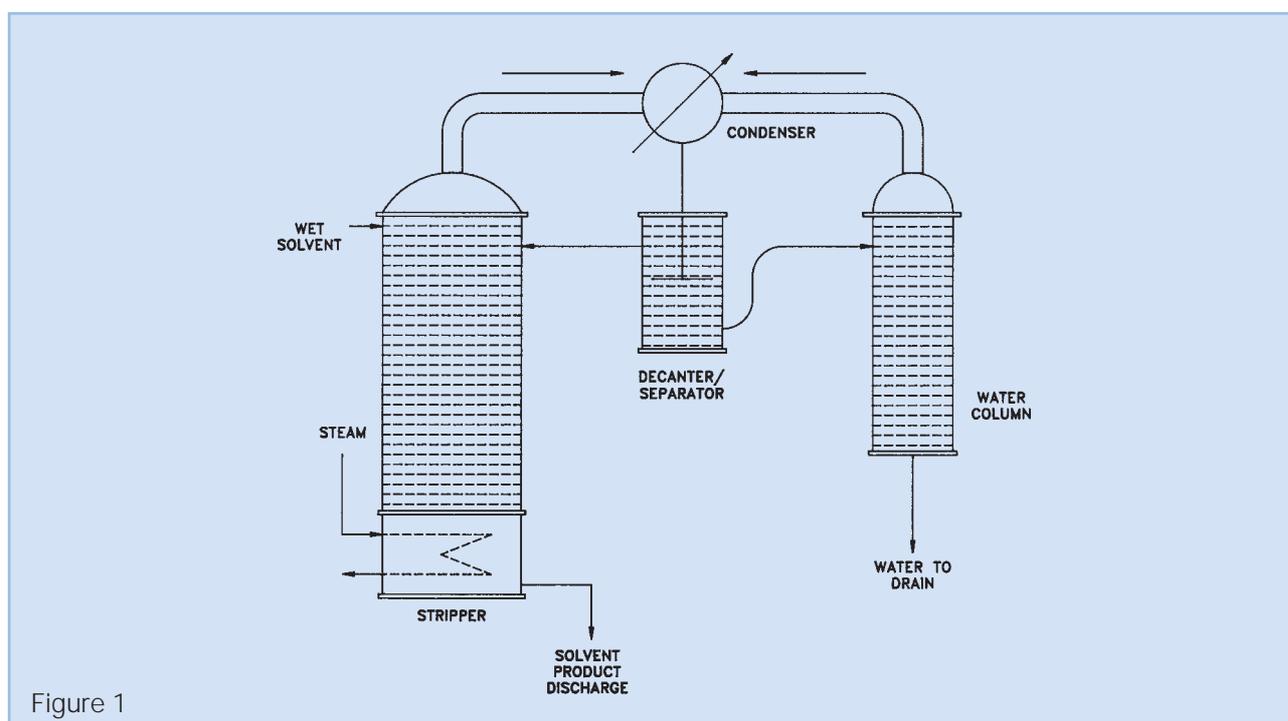


Figure 1

Organic solvents are used in very large quantities, especially as reaction media or for extracting products, in the pharmaceutical, fine chemical and fragrance industries. Solvents can represent a very significant element of the costs associated with any particular product and often need to be reclaimed and recycled. They can also introduce environmental hazards, not only as airborne vapours, but also in the form of waste- and ground-water contamination arising from accidental spillage and other losses. The Servomex Xendos 2500 infrared analyser plays an important role in fulfilling the measurement and control requirements in both the economic and environmental cases.

Solvent quality monitoring

Solvents are generally recovered by either filtration or centrifuging and are often contaminated by residual water. A batch distillation process may be employed to purify the recovered solvent to an acceptable quality for re-use, the end-point of the distillation being determined by the remaining water content (see Figure 1). In some cases, a low percentage level of water remaining is quite acceptable, but in others nearly all the water must be

removed before the solvent is suitable for re-use.

The continuous, on-line measurement of water in solvents can be achieved using a Servomex Xendos 2500 infrared analyser. Fitted with a suitable sample conditioning system, the analyser can be mounted close to the distillation plant to give both a digital display and analogue measurement signal of the solvent's moisture content. It can be calibrated to measure water at the percentage level in a given solvent, operating in the near-infrared region of the spectrum at either 1.4 or 1.9 microns wavelength, or to measure moisture at ppm (parts-per-million) levels using the mid-infrared band at 2.7 microns. Typical solvents monitored include methanol, acetone, tetrahydrofuran and dimethylsulphoxide .

Waste water contamination monitoring

Accidental spillage and plant leaks of solvent may very easily enter the drainage system of a typical plant and contaminate the waste water effluent. In most cases, the effluent treatment plant is able to eliminate fairly low levels of solvent contamination but may not cope with sudden and unexpectedly high levels arising from accidents and leaks. Under these circumstances,

the waste water should be diverted into storage for dilution and eventual processing to prevent high solvent levels causing damage to the treatment processes and serious pollution.

Servomex process infrared analysers are employed in several installations to monitor for waste water contamination. They are configured to measure vapour phase concentrations of specified solvents at vpm (volume part-per-million) levels in the air headspace above the flowing effluent water in the plant's sub-surface drainage system. When a leak occurs and finds its way into the drain, there is an immediate increase in the vapour phase concentration which is very rapidly measured and an alarm raised, enabling suitable corrective action to be promptly taken. This technique is suitable for simple hydrocarbons, chlorinated solvents and others immiscible with water, as well as soluble solvents (such as methanol) since an equilibrium vapour phase concentration can still be detected in the confined headspace.

Virtually all solvents have significant infrared absorbances and the infrared analysers can be configured to suit any measurement wavelength. In addition, groups of solvents (such as the hydrocarbons) can be summed together using the common absorption bands.

Other applications

a) Safety monitoring of oxygen concentration in centrifuges

The presence of high levels of solvent vapours inside centrifuges used to recover product or intermediates poses a serious explosion risk which is avoided using nitrogen purging to reduce the oxygen concentration to a safe level. To minimise nitrogen wastage but still ensure safety, the Servomex 1100A paramagnetic oxygen analyser with a solvent resistant cell is used to measure residual oxygen levels and provide a safety interlock with the centrifuge control system.

b) Solvent recovery and incineration systems

Recovery of solvent vapours arising from various processes can be achieved using carbon bed adsorbers. The efficiency of these can be maximised by monitoring solvent vapour concentrations using the Servomex Xendos 2500 infrared analyser. If waste solvents are being incinerated for energy recovery, monitoring of the flue gas emissions may be necessary. Servomex can also supply fully integrated emission monitoring systems.

Servomex has a policy of constant product improvement and therefore reserves the right to change specifications without notice.



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c) Caustic (sodium hydroxide) scrubbing of acidic gases

Acidic and usually toxic gases (such as hydrogen chloride or sulphur dioxide) may be used as reactants in pharmaceutical processes or may be given off as byproducts. In plants equipped with caustic scrubbing towers to remove these gases prior to venting to atmosphere, adequate control of the sodium hydroxide solution strength must be maintained. The Servomex Xendos 2500 analyser can be used to provide a truly linear measurement of sodium hydroxide concentration to ensure reliable operation.

Servomex analysers and systems

The Servomex Xendos 2500 infrared analyser is a versatile and highly robust instrument suitable for operation in all types of process environment with full certification for hazardous area use. It is highly resistant to shock and vibration, and can tolerate significant levels of sample cell contamination while still making reliable measurements. The analyser can be configured to measure a wide variety of gases and vapours, as well as water in organic liquids. It is fully equipped with facilities for continuous unattended operation and is easy to use.

The Servomex 1100A oxygen analyser employs the well-proven paramagnetic cell design which is fast and accurate and virtually unaffected by other gases in the sample. Since it uses a physical measurement technique, there are no chemicals or electrolyte to replace and maintenance requirements are low. The instrument is robust and suitable for installation in hazardous areas.

Servomex can supply analysers together with all of the appropriate sample handling and mounting hardware to meet many different applications in the pharmaceutical and allied industries.

Reference information:

Xendos 2500 Series Process Gas Analyser	TDS 2500
Safety Monitoring Of Oxygen Concentrations In Centrifuges	AP05
Continuous Monitoring Of Biological Reactors	AP06
Measurement Of Caustic Solution Strength	AP10
Trace Moisture Measurement	AP17
The Measurement of Water by Infrared	AP23
Monitoring The Efficiency Of Solvent Recovery Plant	AP21



Servomex Group Limited:

Servomex International Limited, Jarvis Brook, Crowborough, East Sussex, TN6 3DU, England

Servomex B.V., Stephensonstraat 20, 2723 RN Zoetermeer, Netherlands

Servomex S.A., 8 Rue Proudhon, B.P. 50, 93212 St Denis La Plaine Cedex, France

Servomex GmbH, Münsterstraße 5, 59065 Hamm, Germany

Servomex Company, Inc., 90 Kerry Place, Norwood, MA 02062, USA

Servomex Asia Pacific Ltd, 5F-4, No. 328 Chang Chun Road, Taipei, Taiwan

CNTIC-Servomex Technical Service Centre, No.2 Sheng Gu Zhuang, Chaoyang Area Beijing, China, PO Box 9821, Beijing, Post Code 100029

Global email: info@servomex.com

☎ (44) 1892 652181. Fax: (44) 1892 662253
 ☎ (31) 79-346 42 42. Fax: (31) 79-342 08 19
 ☎ (33) 1 49 46 22 50. Fax: (33) 1 48 20 63 58
 ☎ (49) 23 81 68 82 13. Fax: (49) 23 81 68 81 75
 ☎ (1) 781-769-7710. Fax: (1) 781-769-2834
 ☎ (886) 2-2718 0031. Fax: (886) 2-2712 0653
 ☎ (86) 10-64427984. Fax: (86) 10-64418694

Website: <http://www.servomex.com>