Gas Analysis
Application Note 223

Quadrupole Mass Spectrometry

Data from Fermentation Monitoring

Summary

The FSMS series mass spectrometers offer data from fermentation processes in a form which suits your particular application. For example, in the laboratory data is often required from a single fermentor using a dissolved species probe and Off-Gas probe for analysis. Typical species monitored are shown in the list attached.

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In pilot plant and production scale fermentors, information relating to yield of product is usually of most importance. Here the mass spectrometer is configured with a multiport Off-Gas analysis manifold. Data used will often be for the calculation of the respiratory quotient, carbon production rate and oxygen uptake rate.

The Off-Gas measurement provides the respiratory quotient in a time and space averaged form and with good response time. By comparison data from multiple dissolved species probes are more specific and can provide information about stirring efficiency, aeration and feed distribution within the fermentor. Dissolved gas measurements have the advantage of giving direct information on gas uptake and for dissolved output rates. Hiden Analytical offer systems with either Off-Gas or dissolved species inlets or both. Data presentation and system control is also flexible to suit specific requirements.

Figures 1 and 2 show the progression of oxytetracyclin and erythromycin fermentations with comparison of Off-Gas measurements and dissolved gas measurements (Bohatka et al. 1983 a,b,c; Lloyd et al. 1985).

Figure 3 shows the data from a laboratory experiment in which dissolved O2, H2, and CH4 concentrations are measured in a sample of bovine rumen liquor using a dissolved species probe. (Scott et al. 1985).

Figure 4 shows the effect of O2 on CH4 formation in a sample from an anaerobic digestor (Lloyd & Scott, 1985). Typical applications where FSMS systems are used:

- **Soil Core Analysis**
- **Fermentation Process Analysis**
- **Water analysis in Estuary/River/Reservoir environments**
- **Methane production**

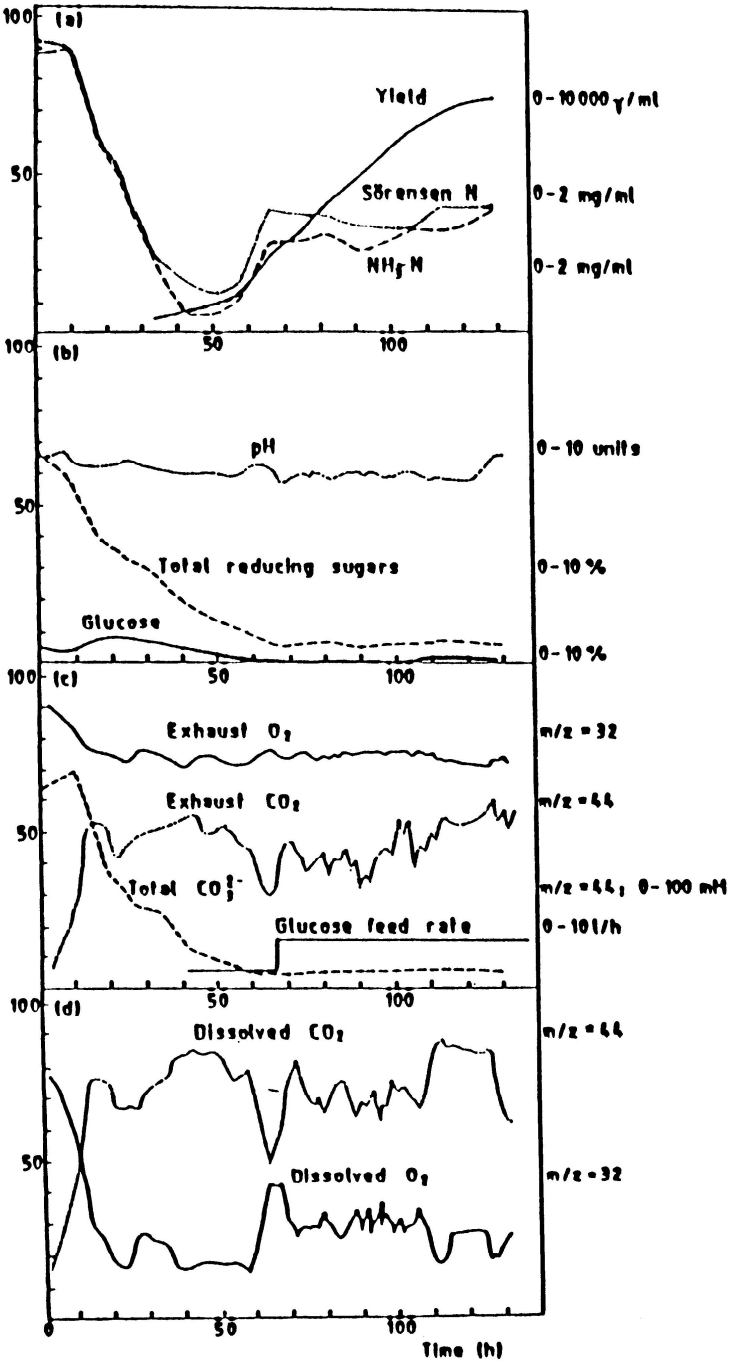

***Figure 1**. Process curves for the oxytetracyclin fermentation. Quadrupole mass spectrometer output signals are in arbitrary units. Lloyd et al. (1985).*

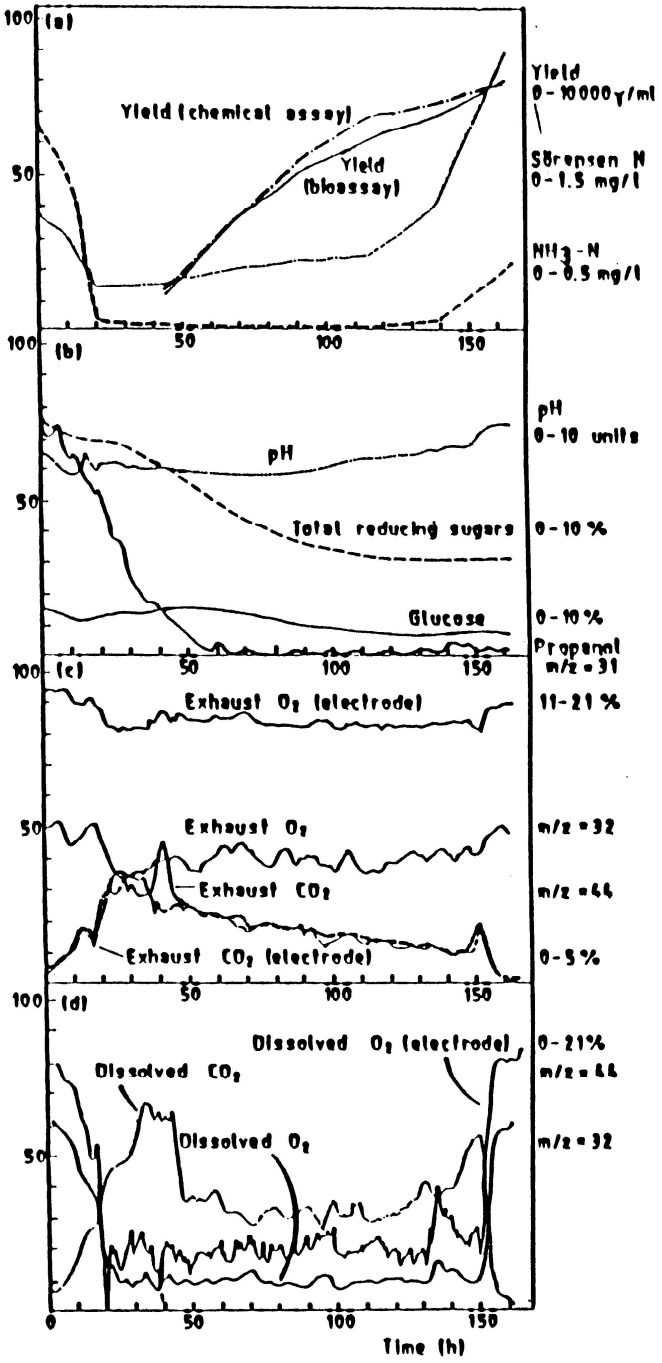

***Figure 2**. Process curves for the erythromycin fermentation. Quadrupole mass spectrometer output signals are in arbitrary units. Lloyd et al. (1985).*

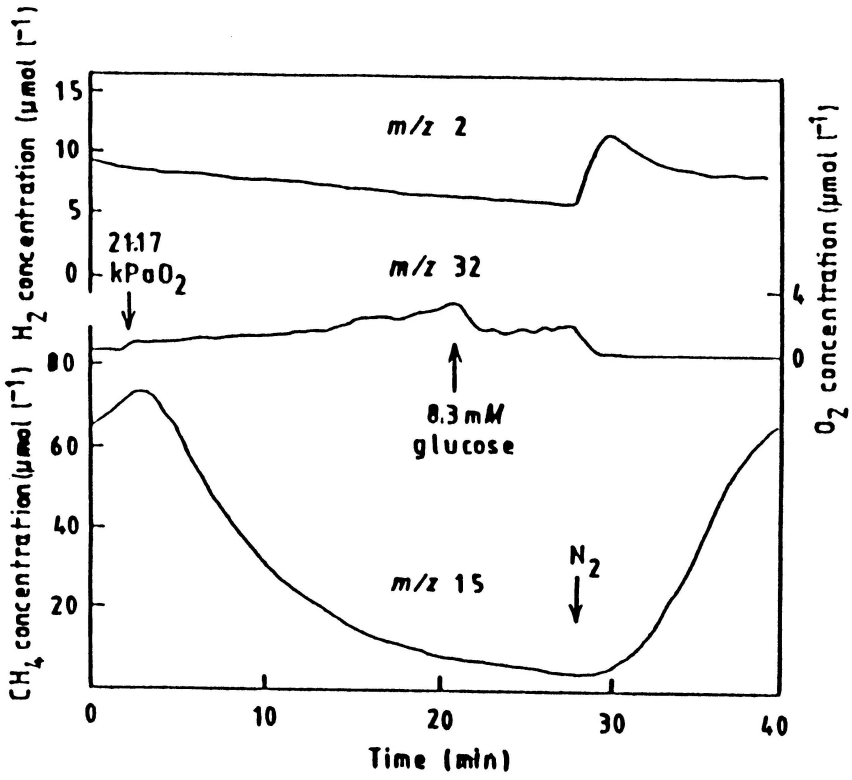

Figure 3. *Effects of O_2 on methanogenesis in a sample of bovine rumen liquor. H_2 was measured at m/z 2, O_2 at m/z 32 and CH_4 at m/z 15. The gas phase was changed from 21.17 kPa O_2 to 101 kPa N_2 (100%) at the time labelled N_2. Scott et al. (1985).*

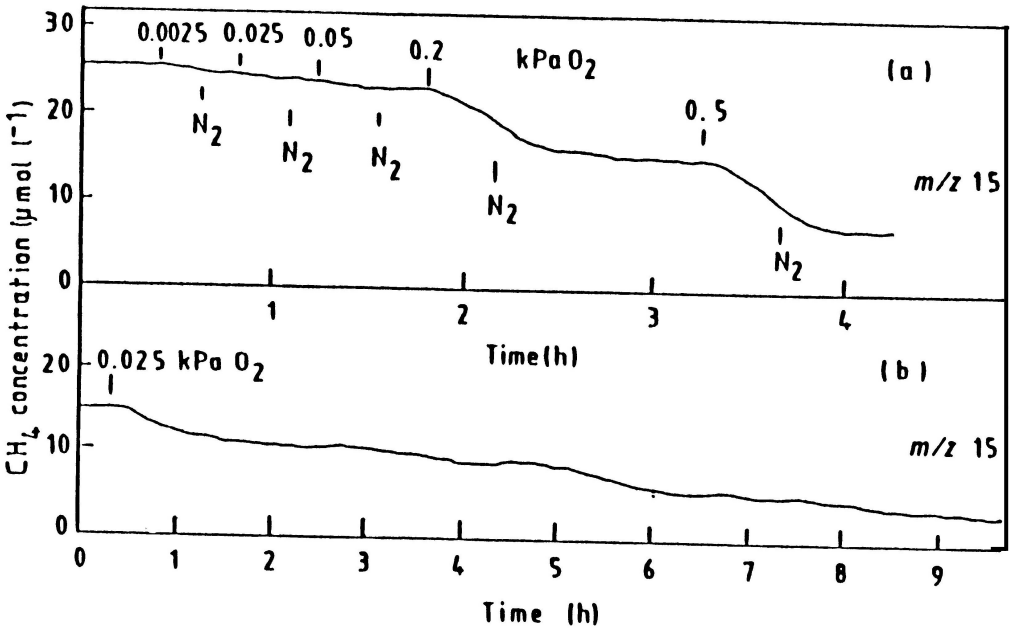

Figure 4*. Effect of O_2 on CH_4 formation in a sample from an anaerobic digester (domestic waste) (a). Effect of various partial pressures of O_2 (b). Effect of longer exposure to O_2. Dissolved H_2 and O_2 were both undetectable. The gas phase was changed from the partial pressure of O_2 indicated to 100% N_2 (101 kPa) at the times labelled N_2. Lloyd and Scott (1985)*

H_2 is measured at m/z = 2. Interference from other species or vacuum background is generally small.

CH_4 is measured at m/z = 15. This is preferable to measurement of the molecular ion at m/z = 16 to minimise possible interference from NH_3 (7% of its largest peak at m/z = 18) and NO, and to exclude contributors from O_2, CO_2, other oxides of nitrogen and water at the latter value. AT m/z = 15, apart from NH_3, the only likely interfering component of fermentation liquors is ethane. No polarographic method is available for measuring methane.

O_2 is measured at m/z = 32. The only possible interference is from H_2S (44% of its largest peak at m/z = 34), or methanol (67% of its largest peak at 31).

CO_2 is measured at m/z = 44. The only possible interference is from N_2O (formed by many de-nitrifiers) and from volatile fatty acids (formic acid has twice the contribution of acetic acid at m/z 44).

H_2S is measured at m/z = 34. No contributions at this value from other low mol. wt. species are evident (Lloyd *et al*. 1981).

CO is measured at m/z = 12 (owing to the C+ peak) rather than at 28. At the molecular ion, peak discrimination from N_2 requires the ability to resolve to m=0.01. CO^+ ions fragmented from CO_2 in the ion source also interfere. At m/z = 12 in the absence of hydrocarbons, and after removing CO_2 by absorption in a sampling line, CO can be determined above a lower limit of about 1% using argon carrier gas (Bohatka *et al*., 1980; Lloyd *et al*., 1982a).

N_2 is measured at m/z = 14 has been used to measure cyanobacterial nitrogen fixation (Jensen *et al*., 1981). Choice of this value minimises possible interferences from acetylene, ethylene, ethane, carbon dioxide, methanol and volatile fatty acids. Methane, ethylene and oxides of nitrogen also have significant contributions at m/z = 14. In the absence of these components N_2 is a useful calibration standard in aerobic systems.

Ar, measured at m/z = 40, is a useful internal standard in aerobic systems; no other gases interfere at this amu.

Methanol is measured at m/z = 31 (Reuss *et al*., 1975; Weaver & Abrams, 1979) through a silicone membrane directly from solution ethanol, isopropanol and acetic acid may interfere.

Acetaldehyde is measured at m/z = 29, but ethanol (Heinzle *et al*., 1983) methanol, isopropanol, acetone, acetic acid and formic acid all have contributions at this mass number.

Ethanol is measured at m/z = 31, 45 or 46. Methanol, isopropanol and acetic acid may interfere at m/z = 31, and acetaldehyde and formic acid at the higher mass numbers.

Formic acid is measured at m/z = 46; acetaldehyde or ethanol may interfere.

Propanol is measured at m/z = 59 or 60. Acetone, n-butanol, and butyric acid.

Mass spectra for these compounds are shown in Figure 5.

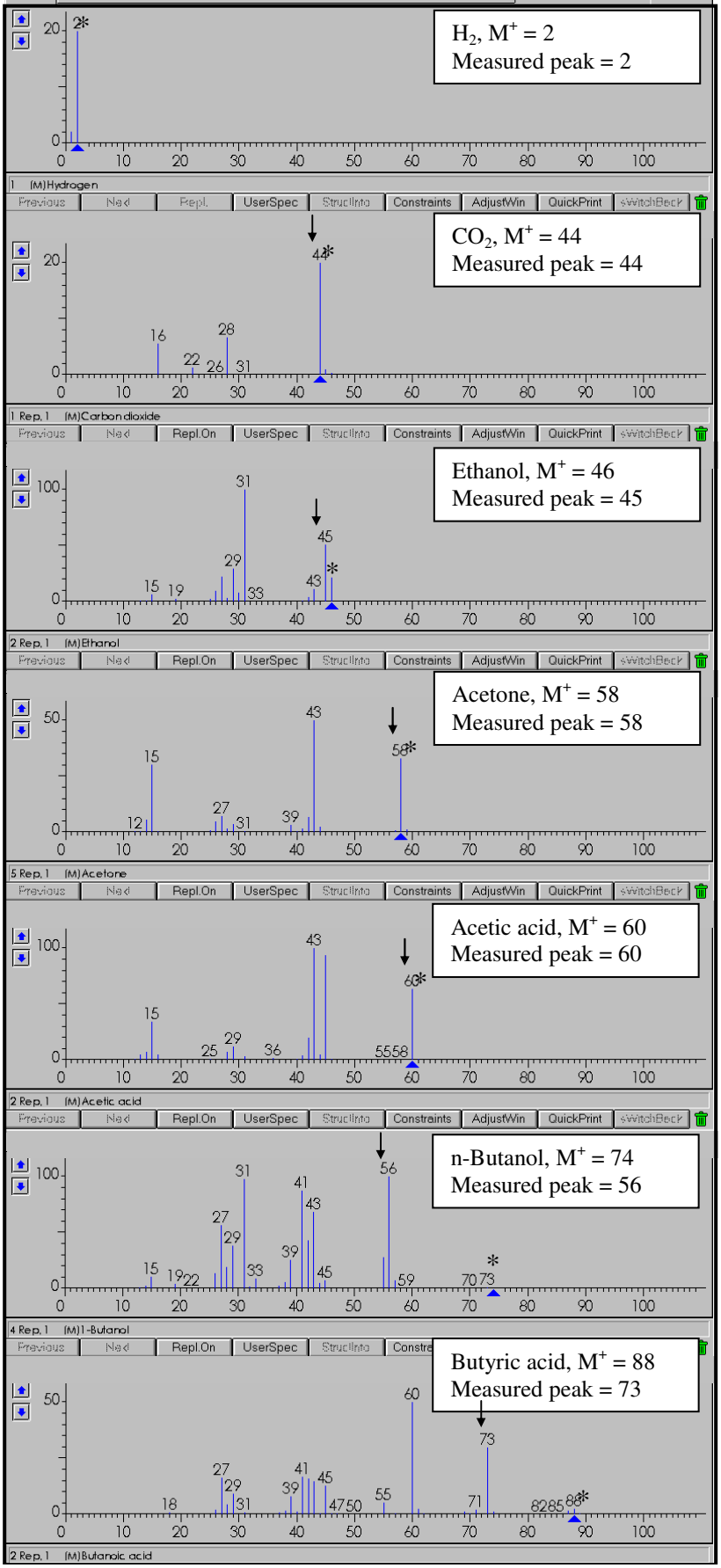

Figure 5. *Mass spectra of products of the acetone-butanol fermentation. The spectra are normalised to the largest peak of each spectrum set to 100. The m/z value of the molecular ion is indicated (*), as is the m/z value of the peak chosen for calculations (arrow). Doerner et al. (1982).*

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