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CHARACTERISTICS, SELECTION AND USE OF RESIDUAL GAS ANALYZERS

by William W. Hultzman

Lewis Research Center

Cleveland, Ohio

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SUMMARY

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Some of the more important characteristics of residual gas analyzers utilizing the mass spectrometer principle of operation are described. Discussion of many of the commercial models now available is based on actual evaluation of, or experience with, instruments currently in use at Lewis Research Center, as well as on manufacturers' published literature. The selection and use of a residual gas analyzer are discussed by considering such performance factors as sensitivity, minimum detectable pressure, mass range coverage, resolution, and mass spectra scan time. Also discussed are operating features including reliability, flexibility, convenience, and analyzer tube envelope construction.

Author

INTRODUCTION

Many types of residual gas analyzer utilizing the mass spectrometer principle are now commercially available. Although principles of operation vary somewhat, the analyzers discussed herein all ionize gas molecules by electron impact and separate the ionized molecules according to their mass-to-charge ratio. All the analyzers are attached directly to vacuum systems and provide instantaneous readout, as distinguished from batch analysis.

Table I lists some of the research projects at the Lewis Research Center which currently utilize mass spectrometers.

TABLE I. - RESIDUAL GAS ANALYZERS IN USE AT LEWIS RESEARCH CENTER

Research area	Partial-pressure range, torr	Number of instruments
Solar simulation	10^{-14} to 10^{-6}	2
Lubrication	10^{-12} to 10^{-5}	5
Surface physics	10^{-11} to 10^{-5}	4
Materials	10^{-10} to 10^{-8}	2
Instrumentation	10^{-10} to 10^{-4}	6
Electric propulsion	10^{-8} to 10^{-5}	2
Energy conversion	10^{-9} to 10^{-6}	5
Liquid metals	10^{-9} to 10^{-6}	2
SNAP testing	10^{-7} to 10^{-5}	1
Centaur testing	10^{-6} to 10^{-4}	1

Although most of these spectrometers are magnetic-sector types, the nonmagnetic quadrupole type instruments are likely to become more popular.

The requirements of a vacuum system determine the choice among the many instruments now available. The following are some of the important instrument characteristics to be considered in such a selection:

(1) Performance factors
  (a) Sensitivity
  (b) Minimum detectable partial pressure
  (c) Maximum operating total pressure
  (d) Mass range
  (e) Resolution
  (f) Mass spectrum scan time
(2) Operating features
  (a) Reliability
  (b) Flexibility
  (c) Convenience
  (d) Construction

PERFORMANCE FACTORS

Table II lists some of the performance factors of various commercial instruments now available. Those which have been evaluated at Lewis by the author are indicated.

Sensitivity

Sensitivity of a residual gas analyzer, as usually defined, is proportional to the slope of the graph of ion current against partial pressure and varies with the species of gas analyzed. The sensitivity is usually measured as amperes per torr of nitrogen at the specified normal operating emission current (normally 1 μA to 1 mA).

The nitrogen sensitivity of instruments using Faraday-cup ion collectors is generally of the order of 10^{-4} ampere per torr. Sensitivity becomes nonlinear above approximately 10^{-4} torr, as in ion gages which measure total pressure, and is a result of space charge effects, small mean free paths, ion-molecule interactions, etc. Low-pressure measurements are not limited to the same extent as are measurements in total-pressure ion gages. Geometry and shielding of electrodes minimize the effect of X-ray or other residual currents.

To increase sensitivity, many instruments are now using electron-multiplier ion

TABLE II. - PERFORMANCE FACTORS OF SOME COMMERCIAL

RESIDUAL GAS ANALYZERS

[Except as noted, data are based on manufacturers' published literature.]

Gas analyzer	Minimum detectable nitrogen pressure, torr		Mass range, amu	Resolution at 50 percent of peak height, $(M/\Delta M)_{0.5}$
	Electrometer	Electron multiplier		
Magnetic deflection				
Aerovac AVA-1 (60°)[a]	10^{-9}	------	2 to 70	45
C. E. C. 21-612 (180°)[a]	$<10^{-9}$	------	2 to 80	40
C. E. C. 21-614-1 (cycloidal)	$<10^{-10}$	------	2 to 200	200
C. E. C. 21-614-2 (cycloidal)	$<10^{-11}$	------	2 to 200	80
G. E. 22 PC-110 (90°)[a]	10^{-10}	$<10^{-13}$	1 to 300	130
Nuclide 21-90-MB (90°)	------	10^{-14}	1 to 100	100
Veeco GA-4 (60°)[a]	$<10^{-10}$	$<10^{-13}$	2 to 300	150
Omegatron				
Edwards	10^{-10}	------	2 to 200	------
Leybold[a]	10^{-9}	------	1 to 250	1000/M
Sloan OMS-2	10^{-10}	------	2 to 50	------
Vacunetics	10^{-10}	------	2 to 86	1000/M
Electrostatic and radiofrequency				
Abcor WB-7 (Bennett)	10^{-9}	------	1 to 250	40
Leybold Farvitron[a]	10^{-8}	------	1 to 250	20
Leybold Topatron (Bennett)	10^{-8}	------	2 to 100	30
Time of flight				
Bendix 17-210 (modified)[a]	------	$<10^{-12}$	1 to 400	50
Quadrupole or monopole				
Atlas AMP-3 (quadrupole)	10^{-10}	$<10^{-12}$	1 to 100	[b]100
G. E. 22 PC-160 (monopole)	------	$<10^{-13}$	1 to 600	[b]600
Ultek 200 (quadrupole)[a]	------	10^{-14}	1 to 500	[b]500
Varian 974 (quadrupole)	$<10^{-10}$	$<10^{-12}$	1 to 250	5 to 100

[a]Evaluated by the author.

[b]At high end of range.

detectors. At Lewis, sensitivities from 0.5 to over 100 amperes per torr have been measured for nitrogen for the time-of-flight, magnetic-sector, and quadrupole spectrometers.

Multiplier ion detectors vary more in sensitivity than Faraday-cup collectors. Multiplier current output is affected by contamination of electrodes, ion accelerating energy, ion molecular structure and mass number, and multiplier saturation at higher pressures.

If pulse-counting techniques are employed, rather than the more common current amplifiers, sensitivity variations are reduced. Use of electron multipliers allows reduced input time constants and, therefore, faster response time. Bandwidth remains relatively wide.

Minimum Detectable Partial Pressure

The lowest detectable partial pressure is herein defined as the nitrogen partial pressure represented by an ion current signal twice the peak amplitude of the noise of the system. Although replacing a Faraday-cup detector with an electron multiplier results in a theoretical millionfold increase in gain, the accompanying increase in noise level results in a net decrease in minimum detectable pressure by a factor of 10^{-3} to 10^{-4}, as shown in table II.

The ratio of minimum detectable pressure to total pressure, although not often specified, varies from 1 part per hundred for a space-charge limited instrument like the Farvitron to more than 1 part per million (under favorable conditions) for some other types of gas analyzers equipped with electron multipliers.

Maximum Operating Total Pressure

Maximum operating total pressure is usually about 10^{-4} torr, being limited by nonlinear output, as previously discussed, and by possible damage to the thermionic filament. However, instruments with electron-multiplier ion detectors are preferably limited to pressures below 10^{-5} torr because of multiplier current saturation and because of the danger of multiplier contamination from hydrocarbons, water, and other impurities.

Mass Range

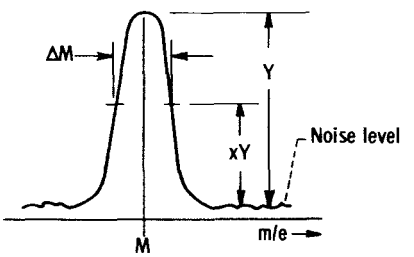
The mass range of the instruments discussed herein varies from 2 to 50 atomic mass units for some, to 1 to 600 atomic mass units for others. Although the term atomic mass

unit has become a conventional designation, the physical quantity actually represented is the ratio of mass to single ionic charge m/e.

Resolution

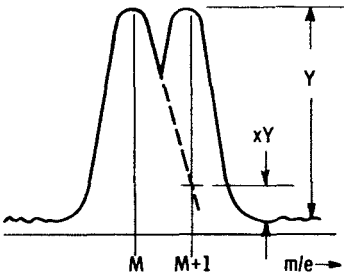
The resolution of a mass spectrometer is a figure of merit that defines the ability to separate mass peaks adequately. Various means of describing comparative resolving powers (or measures of resolution) are illustrated in figure 1. These are not all just different ways of measuring the same performance parameter. Each of the measures actually describes a different feature of performance, although some are so slightly different that the conversion from one measure to another involves little approximation.

The principal types of measures are obtained as follows:



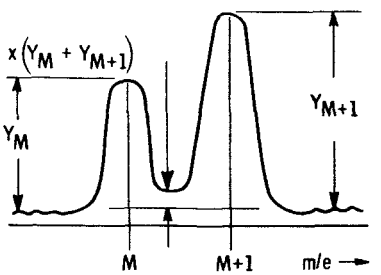
Manufacturer	Type of instrument	Fraction of peak height, x
G. E.	90°	0.01
Leybold	Farvitron	.5
Leybold	Omegatron	.01, .5
Leybold	Topatron	.1, .5
Varian	quadrupole	.5

(a) Measurement of single peak width.



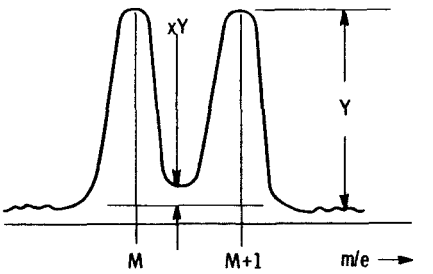
Manufacturer	Type of instrument	Fraction of peak height, x
C. E. C.	cycloidal	0.01
Bendix	T. O. F.	.01, .015, 1.0
G. E.	90°	.01

(b) Measurement of cross contribution between adjacent peaks.



Manufacturer	Type of instrument	Fraction of peak height, x
Atlas	quadrupole	0.05
G.E.	monopole	.1
Nuclide	90°	.002
Sloan	Omegatron	.1
Veeco	60°	.01

(c) Measurement of valley between adjacent peaks of unequal height.



Manufacturer	Type of instrument	Fraction of peak height, x
Abcor	r-f	0.35
Bendix	T. O. F.	0, 1.0
C. E. C.	180°	.02
Ultek	quadrupole	.1

(d) Measurement of valley between adjacent peaks of equal height.

Figure 1. - Some manufacturers' methods of describing resolution.

(1) By measurement on a single peak at mass number M. The resolution is given as $M/\Delta M$, where ΔM is the width (in mass units) at a stated fraction x of peak height Y, usually one-half (fig. 1(a)).

(2) By measurement on two peaks separated by one mass number. The mass number for stated cross contribution is the highest mass number at which one peak contributes a given percentage (generally 1 percent) to the height of the adjacent peak (fig. 1(b)). When not actually specified, it is tacitly assumed that both peaks are of the same height.

(3) By measurement on two peaks (Y_M and Y_{M+1}) separated by one mass number. The mass number for unit resolution is that mass number at which the height of the valley between peaks is a stated fraction x of the sum of the two peak heights (fig. 1(c)). Sometimes this fact is not mentioned in the description and, instead, both peaks are stated to be of the same height (fig. 1(d)). The definition of figure 1(c) is more general than that of figure 1(d), and also more realistic, since the user rarely can arrange to have both peaks of equal height.

The measures used by different manufacturers are indicated, in figure 1, to the extent that they are known or understood by the author. Some manufacturers give more than one measure for the same instrument in order to clarify performance. Others provide an incomplete description, so that the author has had to guess at the remainder of the description.

In order to provide some common basis for comparison, the data listed in the column headed Resolution in table II (p. 3) were based on measure (1). The numbers represent actual measurements for those instruments which have been evaluated at Lewis; for the other instruments, the numbers are based on the manufacturer's published literature. Where the manufacturer uses a measure other than (1), the author, from his experience with evaluated instruments, has estimated the equivalent value in terms of measure (1) by establishing a crude empirical relation between measures (1), (2), and (3).

As the ratio $M/\Delta M$ remains relatively constant for magnetic, radiofrequency or time-of-flight instruments, and some quadrupole instruments, the ability to separate adjacent peaks varies inversely with mass. Resolution ($M/\Delta M$) for omegatron-type instruments, however, is not constant and varies inversely with mass. Some quadrupole and monopole instruments have approximately constant ΔM, and, therefore, the ability to separate peaks remains constant. Typical values of ΔM for the latter instruments range from 0. 5 to 5 atomic mass units. However, a front-panel control, usually available, permits trading resolution for sensitivity.

The electrostatic and radiofrequency type instruments, which do not require magnets, have not been widely used in this country up to this time, presumably because of their comparatively low sensitivity, low resolution, and glass construction. However, they may become more popular as the residual gas analyzer is adopted for routine engineering

use as a process monitor.

Maximum resolution and maximum accuracy of peak-height indication can be obtained only when the rate of mass scan, expressed as atomic mass units per unit time, is small compared to the reciprocal of the overall time constant of the amplifying and recording system.

MASS SPECTRUM SCAN TIME

Time required for completely scanning mass ranges varies from 10^{-4} second per spectrum for pulsed time-of-flight instruments, to milliseconds for the Farvitron and instruments with electron multipliers, and to minutes for instruments with electrometer ion current amplifiers.

OPERATING FEATURES

Table III lists some of the operating features considered important in selecting a mass spectrometer.

Reliability

Reliability of operation has varied widely among instruments used at Lewis. Herein, reliability refers to mechanical and electrical dependability, as well as repeatability and accuracy of measurements. Some instruments of certain manufacturers have a record of frequent mechanical and electrical breakdowns; others have a record of continuous trouble-free service. Flexibility and sensitivity are not correlated with reliability. Poor mechanical reliability is generally associated with inadequate attention to details of design and construction. Poor electrical reliability may be associated with poor choice of components, with inadequate voltage or frequency regulation of power supplies, or with highly complex circuitry in which the mere number of components is sufficient to increase the probability of a failure. Some types of spectrometers inherently require closer voltage or frequency regulation than other types; the time-of-flight, quadrupole, monopole, and radiofrequency types require very precise voltage regulation, and the last three of these also require very precise frequency regulation.

The following are some of the measures that may be applied to calibration stability:

(1) Ability to monitor a single mass continuously, without frequent manual readjustment of controls

TABLE III. - SOME OPERATING FEATURES OF VARIOUS COMMERCIAL MASS SPECTROMETERS

[Data obtained from manufacturers' published literature; X indicates feature is present.]

Operating feature			Spectrometer										
			Aero-vac	C. E. C. 180°	G. E. 90°	Veeco	Omega-tron	Farvi-tron	Bendix	C. E. C. cycloi-dal	Nuclide	RF (Bennett)	Quadru-pole and monopole
Reliability	Mechanical workmanship[a]												
	Electronic design[a]												
	Calibration stability[a]												
	Circuit complexity				X			X	X			X	X
Flexibility	Presentation	Cathode ray oscilloscope			X	X		X	X				X
		X-Y recorder	X		X								X
	Total-pressure indication		X									X	X
	Adjustability of operating parameters	Full			X		X		X		X		X
		Limited	X	X		X		X		X		X	
Convenience	Bakeout capability	Full	X	X	X	[b]X	X	X			X	X	X
		Limited							(c)	(d)			
	Magnet positioning	Fixed	X			X					X		
		Not fixed		X	X		X	(e)	(e)	X		(e)	(e)
	Range change	Remote		X	X	X	X			X		X	X
		Local	X		X	X							
		None			X			X	X		X		
Construction	Envelope size	Small		X			X	X				X	
		Medium	X		X					X			X
		Large				X					X		
		Very large							X				
	Envelope material	Metal	X	X	X	X			X	X			X
		Glass			X		X	X				X	
	Ion source	Nude	X		X	X			X		X	X	X
		Tubulated		X	X	X	X	X		X		X	X

[a]See text for discussion of reliability.

[b]Bakeout limited to 300° C with electron multiplier.

[c]Bakeout limited to 200° C.

[d]Bakeout limited to 250° C.

[e]No magnet required.

(2) Constancy of proportionality between ion current and partial pressure over several decades of pressure (The Farvitron analyzer generally operates in a space-charge-limited condition, in which it produces a spectrum whose total area is constant. If a new gas is then injected into the chamber or removed from it, the heights of all other peaks will also change; however, the relative heights usually will correctly represent the relative partial pressures.)

(3) Ability of magnetic-deflection instruments to maintain constant magnet orientation relative to the electrodes, and, in the case of electromagnets, to maintain constant magnetizing current (When mass ranges are changed by shunting a permanent magnet, sensitivity is usually adversely affected.)

(4) Freedom from susceptibility to contamination which may contribute excessive background noise or excessive electrical leakage between closely spaced electrodes

Flexibility

Flexibility herein refers to ion detectors and readout devices available for a particular instrument, types of operating controls, and other unique features.

As shown in table II (p. 3), many instruments incorporate electron multiplier ion detectors to increase sensitivity over the Faraday-cup detector - electrometer combination and are designed so that the detectors are interchangeable. However, contamination becomes more serious because bakeout temperature of multiplier detectors is often limited. Thus, the increased sensitivity is often obtained at the cost of frequent recalibration and cleaning, or other treatment. Many manufacturers also warn against prolonged exposure of their multipliers to the atmosphere.

Since stray magnetic fields affect multiplier gain, it is impractical to provide electron-multiplier detectors for the cycloidal and omegatron spectrometers because the ion collectors are immersed in the magnetic field. The Veeco instrument provides magnetic shielding for its electron multiplier and thereby increases gain by a factor (30 to 100) that is a function of mass number.

Faraday-cage detectors are thus preferable, in most applications, when pressures exceed 10^{-9} torr, or where fast response time is not required.

All instruments, other than the Farvitron, provide for panel-meter readout and for strip-chart recording of ion collector current as a function of time. The Farvitron and all instruments that have electron multipliers provide for cathode-ray-oscilloscope display. The Veeco analyzer uses the sawtooth signal from the oscilloscope to produce the mass sweep and thereby provides high flexibility in scan time and mass range selection. Veeco's three-segment linear scale, which resembles a logarithmic one, permits a sig-

nal of 0.05 percent of full scale to be detected at any one amplifier gain setting. The General Electric instruments have a five-decade logarithmic scale. Bendix circuitry permits monitoring five channels continuously while the spectrum is being scanned; alternately, as many as six peaks can be monitored continuously if scan is not monitored.

Some instruments provide output voltages that can be applied to X-Y recorders or to oscillographs to produce a direct time-independent plot of equivalent partial pressure as a function of mass.

In addition to total-pressure indication by the instruments identified in table III, the Aerovac, Bendix, and Varian instruments provide overpressure protection circuits.

Limited or fixed voltage and current adjustments are desirable in an instrument intended for routine field operation. Operational reliability is often higher when there is a minimum number of controls. On the other hand, flexibility is desirable in instruments intended for research. The G. E. instrument provides for magnetic scanning as well as for scanning by variation of ion accelerating voltage. Magnetic scanning provides a more open mass scale and less reduction in sensitivity at higher masses because of constant ion accelerating voltage.

Convenience

Convenience here refers to the procedure required in routine instrument operation.

Mass spectrometers require a full bakeout (at least 400° C) in order to make their spectrum representative of vacuum-chamber conditions. After sufficient contamination, electron multipliers ordinarily must be baked to restore sensitivity. The best procedure for accomplishing this baking has yet to be learned, both by manufacturers and users, because it is presumably affected by the nature of the contamination. To date, the author has found, in his limited experience, that a manufacturer's instructions, when followed literally, do not always produce a full restoration of gain.

Magnet position is very critical in magnetic-deflection instruments; in those instruments where fixed stops are not provided, the user must devote considerable effort to providing reliable means for restoring the magnet to its original position after an operation such as removal for bakeout. The omegatron, with its glass tube and very powerful magnet, provides an operational hazard because a metallic object attracted to the magnet may break the glass tube.

The Aerovac spectrometer requires a manual substitution of magnets to change mass range. The G. E. instrument can cover almost the entire mass range with one magnet, at the expense of resolution. Full resolution can be achieved if two magnets are used or if a single electromagnet is used; the latter, of course, is controllable remotely. The Veeco instrument offers both manually and pneumatically changed magnet shunts; the

TABLE IV. - MASS RANGES FOR SOME COMMERCIAL SPECTROMETERS

Spectrometer	Mass range, amu
Aerovac	
Small magnet	2 to 11
Large magnet	11 to 70
G. E.	
Small magnet	2 to 200
Large magnet	12 to 300
Electromagnet	1 to 300
Veeco	
Manually changed shunt, shunt in	2 to 50
Manually changed shunt, shunt out	12 to 300
Pneumatically shunted magnet, shunt in	2 to 12
Pneumatically shunted magnet, shunt out	10 to 250

manually shunted magnet, although providing a mass range up to 50, does so with reduced sensitivity. The ranges obtainable for the spectrometers just discussed are listed in table IV.

Construction

Envelope size affects handling and mounting; it also affects outgassing ability if the ion source is not nude. The Bendix spectrometer is preferably operated with a separate high-vacuum pumping system to maintain a high vacuum in the flight tube.

As additional metal analyzer tubes become available, the engineering application of residual gas analyzers becomes more feasible than it was when only glass tubes were obtainable.

As in the case of the tubulated ion gage, the limited conductance of any tubulation and, even more importantly, any limitation on the bakeout capability of the tubulation and of the envelope may seriously affect the accuracy of gas analysis. On the other hand, the dangers of mechanical damage and of source contamination are increased when a nude gage is used. In many cases, for instance, the Ultek guadrupole instrument, the distinction between nude and tubulated is not sharp, since the spectrometer may be inserted to various depths into the vacuum system. The omegatron and the C. E. C. diatron and cycloidal spectrometers, which have ion sources immersed in the magnetic field, are not easily adaptable to a nude construction; nevertheless, in the Nuclide instrument the entire spectrometer, including the magnet, is immersed in the vacuum chamber.

CONCLUDING REMARKS

Comments have been detailed on those spectrometers which have been systematically evaluated at Lewis. Comments on other spectrometers have been less detailed because they have depended on the findings of users whose primary interest was not in instrument evaluation. The techniques of application of partial-pressure analyzers are just being learned, and this learning is accelerated as the diversity of applications increases.

Since instrument development is proceeding very actively, the descriptions of instruments presented in this report may require modifications in view of newer developments by spectrometer manufacturers.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, May 3, 1966.

NASA-Langley, 1966 E-3369

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