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**MASS SPECTROMETRIC DETERMINATION OF
ISOTOPIC RATIO WITH VARYING ION BEAM
INTENSITY**

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WITH VARYING ION BEAM INTENSITY

Introduction

In the comparison of ion beams by vacuum tube or other electrometer measurements, the stability of the ion beam intensity is critical. The steps necessary to achieve satisfactory stability account for most of the total time required for assay of a sample. It would, therefore, be desirable to find a method of measurement which would be independent of the stability of the ion beam. The alternate pulse comparator described in this report permits direct comparison of two ion beams even though their intensity drifts or fluctuates. It is necessary only that the ratio between the two beams be constant. The apparatus was constructed as a modification of the standard Hier mass spectrometer which has been used extensively for isotopic ratio determination.

Apparatus

The comparator utilizes a cylindrical commutator which is immersed in an oil bath and is motor-driven. This commutator performs two functions. First, it selects isotope beams A and B so that they fall alternately on the collector plate. Second, it sensitizes the galvanometer network during the central 120° of each 180° half cycle so that the alternate beams are received with opposite polarity. That is, the network is sensitized 30° after the incidence of beam A upon the collector and desensitized 30° before the beam is removed. It is again sensitized, this time with opposite polarity 30° after the incidence of beam B, and desensitized 30° before the change back to beam A. The square wave impulses received by the galvanometer are plotted in Figure 1 for three different beam ratios. The effect of an isotopic ratio other than 1:1 is to cause a deflection of the galvanometer in the favored direction. At a commutator frequency of greater than 15 cycles per second, the individual pulses are not observed with a critically damped galvanometer of 5 second period. The deflection is a continuous movement. If the galvanometer receives pulses in a 1:1 ratio, variation in pulse intensity will not result in galvanometer deflection and the galvanometer will remain in the zero position for all levels of ion beam intensity. A 1:1 ratio of pulses can be delivered to the galvanometer by permitting the entire weaker isotope pulse to reach the galvanometer while only a fraction of the more intense pulse is admitted. It follows that the isotopic ratio may then be read directly from the potentiometer employed to scale down the more intense pulse to the level of the less intense. When this potentiometer is adjusted for zero galvanometer deflection, a wide variation of ion beam intensity will not displace the galvanometer. The comparator circuit is shown schematically in Figure 2. The high voltage

supply and main control panel, described in report B-6.460.1, are modified as shown. The high voltage supply will follow 1% load changes at 13 cycles per second if the grid condenser of the 6AC7 is reduced to .1 mfd.

It is apparent that the duration of the pulse, as well as its magnitude, will determine the extent of galvanometer deflection. The duration of each pulse is determined by the geometry of the commutator, so it becomes necessary either to adjust the duration of pulse A equal to that of pulse B within the expected limit of error or to make a correction for inequality. The correction is made electrically, since adjustment of the commutator assembly would be difficult and would change with wear. The ratio dials are adjusted for zero galvanometer deflection and the ratio would then be read directly from the dials if the pulse durations were equal. The ratio dials are set and not again changed. Selector C is set to the "Zero-Null R" position. Selector switch A is moved to "Potentiometer Scan" position and the galvanometer brought to zero by changing the settings of the two precision potentiometers which now replace the ion beam as a source of pulse potential. The ratio of these two potentiometer voltages is the ratio of the isotope intensities regardless of equality of pulse duration. The pulse duration correction is essentially constant and small, and a calibration scale for the dials may be readily obtained and expected to remain constant. Thus, repeated reference to the calibration potentiometer is unnecessary. This method of correction also eliminates the necessity of correcting the ratio potentiometer scale for the effects of finite loading introduced by the galvanometer and its shunt network.

Assay Procedure

1. Admit sample to the instrument. Set selector A to isotope A position and adjust voltage control A on the main control panel so that isotope A falls on the collector. This is indicated by deflection of the galvanometer. Repeat for the B isotope. These adjustments are made with selector C in "Deflection" position.
2. Start the commutator. Place selector A in "Scan" position and adjust the ratio dials to bring the galvanometer near to the zero position. Snap selector C to zero position to obtain exact galvanometer zero. Return selector C to "Deflect" position and adjust channel A on main control panel for maximum galvanometer deflection. Repeat for channel B. Adjust the ratio dials to zero galvanometer deflection. Snap selector C to zero position as a check. These adjustments should be repeated through another cycle to find more precise settings of the high voltage and ratio controls.
3. Remove the sample from the instrument. The ratio dial settings indicate directly the mole ratio of the two isotopes. Full advance

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of the ratio potentiometer corresponds to a 1:1 mole ratio. One-half advance represents 2:1 mole ratio, etc. Correction for unequal pulse duration is made as indicated above. The necessity of reversing the high voltage scanning order for samples in which the mole ratio is 1:2, for example, is eliminated by the double pole, double throw selector switch D.

Discussion

The fundamental limitations on the precision of this assay method are reproducibility of pulse duration and phasing in the commutator and the ratio of pulse voltage levels to transients produced by circuit interruptions. At voltage levels of .2 to .5 volts, which are normal in this type instrument, the comparator will determine ratios in the range 1:1 to 1:50, with a precision of .5%. This precision may be improved by an increase in the voltage levels. However, the normal ion current is approximately 10^{-11} amperes, which develops only 5×10^{-4} volts in the input of an electrometer with 50 megohms grid resistance. In tests of the comparator the current level was raised to 10^{-8} amperes for brief periods. Increasing the ion current level in this type instrument is not satisfactory because it results in greatly reduced ion source life. The necessity of obtaining square wave output from a current amplifier operating at low current input, places another limitation upon this comparator method. The amplifier must be of the voltage feedback type. The feedback amplifier in the standard instrument is capable of responding to square wave excitation at 13 cycles per second with sufficiently low order of distortion, particularly since the comparator utilizes only the central 120° of each 180° square wave pulse. The limitation arises in the input where a network of high resistance and distributed capacitance introduces an appreciable time constant. Experimentally, a satisfactory behavior was obtained by reducing the grid resistor to approximately 50 megohms. It appears that the alternate pulse comparator method might have application to a type of spectrometer in which the ion source was a capillary arc or condensed spark, capable of giving high levels of beam current. Since this method removes the necessity of securing beam intensity stability, such sources may prove of utility in spectrometer measurements as well as in mass spectrographic determinations.

Summary

1. An apparatus is described which applies alternate pulses from two ion beams to a galvanometer network. The isotopic ratio is read directly from a potentiometer. The comparison is independent of ion beam intensity.
2. This method is not practical for application to the standard Nier assay instrument because of the low ion currents normally used.

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5. It is suggested that this comparison method may have utility in a spectrometer utilizing a high intensity ion source such as capillary arc or condensed spark.

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ISOTOPE
RATIO
(3)

Figure 1.

8:1

0° 180° 270° 360° 450° 540° 630°

1057

figure 2.