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Negative Ion Spectrometry for Detecting Nitrated Explosives

The problem:

A field analysis instrument package specifically for trinitrotoluene (TNT) has long been needed. The instrument must be capable of rapid sniff-test analyses of the vapors immediately surrounding a parcel or a piece of luggage. It has not been possible to use a portable mass spectrometer directly because other organic materials, such as solvents or perfumes, interfere with this particular type of analysis, making it necessary to first run a vapor sample through a gas chromatograph separation column before making a spectrometric study of the fractions.

The solution:

The ionization procedure in the ion chamber of a mass spectrometer is modified so as to produce mainly negative ions by electron capture. The peaks of such negative ions then are monitored a conventional way. Because of the unique electronegativity which the nitro group imparts to any organic material, it was found that TNT and other nitrated organic materials could be identified directly from a sample sniff inlet stream by a suitably modified mass spectrometer.

How it's done:

Several modifications were made to a mass spectrometer to enable it to perform negative ion mass spectrometry over a wide range of electron energies, negative repeller voltages, and ion source pressures. A specially-designed closed ion source, capable of operation at high sample pressures, is used. It has a closed ionization chamber with holes 0.013 in. (0.033 cm) in diameter in front of the filament and in front of the trap. The ion exit slit size is 0.125 by 0.013 in. (0.318 by 0.033 cm).

The large source magnet, being at ground potential and very close to the ion source, was formerly the origin of frequent high-voltage breakdowns. These breakdowns are even more frequent with negative high voltage than with positive voltage because of a field ionization effect. Thus, the original source magnet was removed and replaced by a smaller magnet which is mounted on the source and is at source potential. The magnet arms are made of Alnico V7, and the yoke and pole pieces are made of Armco steel. The field strength of this magnet is 250 gauss.

The source filament is made from a 0.030-in. by 0.0015-in. (0.076-cm by 0.0038-cm) rhenium ribbon, which is used instead of the standard rhenium wire to enhance the electron emission. This design also decreases any loss of ionizing electrons due to misalignment of the filament assembly relative to the extremely-small electron entrance hole. A Bourdon tube precision pressure gauge, which is connected directly to the ionization chamber, is used for measuring the pressure inside the ion chamber. The vacuum systems at the source and the analyzer have also been replaced by independent pumping systems of considerably higher capacity and lower ultimate pressure.

The voltages to the field plates of the electrostatic sector are normally supplied by a set of dry cell batteries which furnish a nominal potential of ± 270 V. Because of the changes in this voltage due to the aging and depletion of the batteries, considerable attention had to be given to various corrective operations such as magnet repositioning. Thus the batteries were replaced by two transistorized power supplies. The negative accelerating voltage is supplied by a

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separate power supply. The necessary feedback from the electrostatic analyzer decade box to the accelerating voltage is provided by the reference voltage from the decade box. This voltage replaces the voltage originally supplied by the reference tube of the high-voltage supply.

The polarity of the electromagnet current is reversed by activating a relay having two sets of contacts which connect the current supply to the magnet either in the positive or the negative mode. The repeller voltage circuit of the ion source power supply has also been modified. In the negative mode, the repeller voltage range is from +25 to -36 V. The electron voltage range is from 0 to 85 V, and it is measured with a digital voltmeter which is connected between the filament and the source block and which floats on the accelerating voltage.

In the unmodified spectrometer the ion source power supply operates at its maximum power. Hence, when lowering the electron voltage, the emission current decreases because the power supply cannot increase the filament current. This deficiency is corrected by using a more powerful supply with better regulation. Finally, in order to avoid additional wiring, the ΔV meter, essentially a 50- μA meter, has been replaced by a 100-0-100- μA meter. The monitor meter polarity does not have to be reversed because a large part of the 8-eV negative ions will remove electrons from the plate, thereby producing a positive monitor reading.

Experimental work with the modified spectrometer has shown that both TNT, nitrobenzene, and all commonly used commercial explosives are characterized by a predominant peak in the negative mass spectrum at a mass-to-charge ratio of 46. Using this as a characteristic peak, the absolute sensitivity of detection of TNT was found to be 10^3 ions/s per μg TNT per μA electron ionizing current, at an electron energy of 4 eV. At 20° C the vapor pressure of TNT is 0.00075 torr (0.10 N/m²) which is about 10^4 times the necessary pressure for normal analysis in the mass spectrometer.

Note:

Requests for further information may be directed to:

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NASA has decided not to apply for a patent.

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