

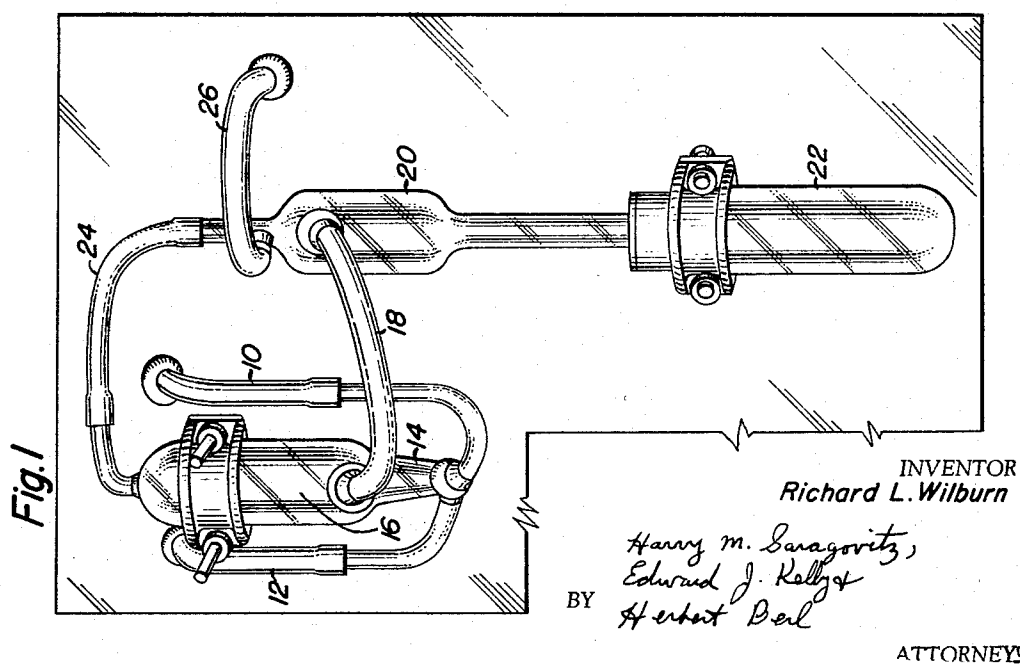
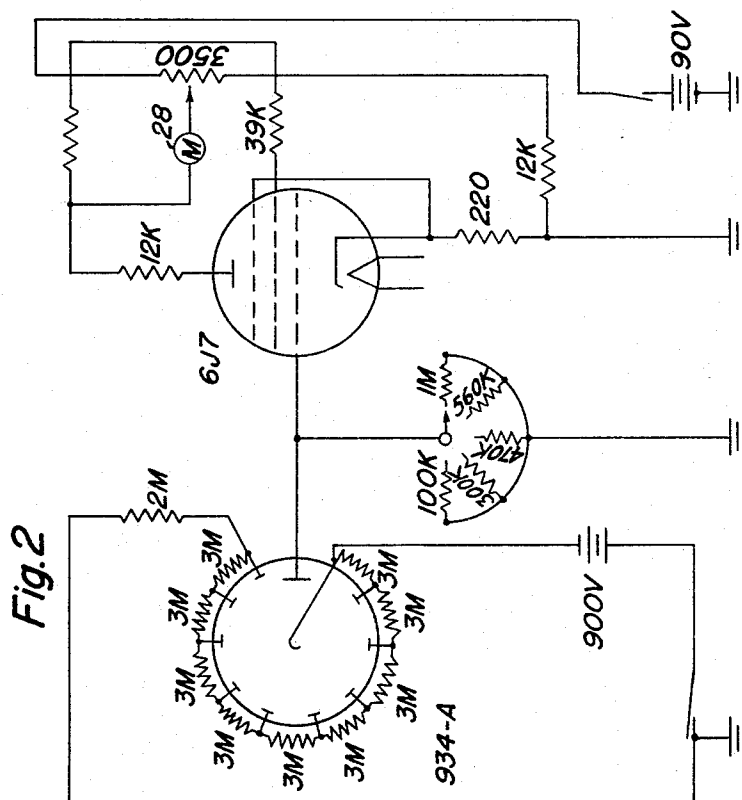
Nov. 22, 1966

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3,287,089

CHEMILUMINESCENCE SAMPLER

Filed May 28, 1964



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3,287,089

CHEMILUMINESCENCE SAMPLER

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Filed May 28, 1964, Ser. No. 371,145

1 Claim. (Cl. 23—254)

The invention described herein may be manufactured and used by or for the Government for governmental purposes, without payment to me of any royalty thereon.

This invention relates to a sampler for the continuous and near instantaneous testing of air for the presence of organo-phosphorous compounds and other oxidants.

These compounds are often dispersed as aerosols for insect control and their presence in the atmosphere should be known in order that the necessary precautions may be taken.

The sampler utilizes the chemiluminescent property of luminol (3-amino-phthalhydrazide) to determine the presence of these oxidants in the air. The air sample is taken in continuously and mixed with the reagent solution. The solution then passes a photomultiplier after which it is exhausted from the sampler. The current from the photomultiplier is amplified and may be displayed on a chart recorder. This invention permits of analysis of concentrations of organo-phosphorous compounds in concentrations as low as 3 gammas per liter of air.

In the drawing, FIG. 1 shows a front view of the apparatus which is mounted on a panel with inlet for air and solution toward the back, and FIG. 2 shows the circuit arrangement for the photomultiplier and recorder.

More specifically, in FIG. 1, 10 is the air intake which projects through the supporting panel to the rear. 12 is the reagent intake which connects to a supply reservoir to the rear of the instrument, 14 is the mixing chamber where air and reagent solution are intermixed after which the solution together with the air sample passes into the transparent viewing cell portion 16. The latter is a clear section of glass tubing, the other portions of the apparatus being coated with an opaque coating. After passing viewing cell 16, the solution passes through tube 18 to sump 20 and passes from the system to storage 22.

Tube 24 is a pressure equalizing connection which serves to equalize the pressure between the sump and mixing chamber. This is necessary to keep part of the solution from being drawn along more than one path, since there has to be a vacuum connection to bring the air sample into the mixing chamber. The solution flows through the system by gravity. Vacuum is applied at 26 and serves to draw the air into the apparatus.

The chemiluminescence reaction of luminol is strongly pH dependent. The more basic the solution up to approximately pH 12.5 the more rapid is the reaction rate and hence the greater the intensity of radiation. The chemiluminescence reaction produces phthalic acid. Therefore, a buffered reagent solution is necessary for good results. The reagent solution for this work contains approximately 5.0×10^{-4} molar luminol, 6.7×10^{-4} sodium phosphate buffer.

The addition of nitrogen-containing bases increases the reaction rate and hence the sensitivity. The use of nitrogen-containing bases such as pyridine also decreases both the time required to reach maximum intensity and the decay time. The composition containing inorganic bases is more sensitive for analysis of ferricyanide, chlorine and other oxidants of this type. If the nitrogen-containing

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base is not used a very slow decay is observed. The major part of this decay is of quite low intensity.

The photomultiplier is placed adjacent the cell at 16 with a mirror behind the cell to retain as much radiation as possible.

The amplifier circuit utilizes a single pentode and a bridge circuit is used as the load for the plate of the amplifier. The vacuum tube and a series resistor makes up one half of the bridge. The resistors, one of which is variable, make up the other half of the bridge between B+ and the ground. The recording milliammeter is connected between the plate of the tube and the midpoint of the two series resistors. This equipment was designed for field use and is operated by batteries.

In this type of power supply, the bridge type of plate load circuit cuts down the effect of the changing B+ on the response of the system.

In operating the sampler, the concentration of luminol should exceed the stoichiometric amount required for the largest expected concentration of sample. The excess should not be large, however, since the reaction suffers from concentration quenching and some sensitivity will be lost.

The variables affecting the sensitivity are air flow rate, solution flow rate and volume of the mixing chamber. The higher the air flow rate, the lower the solution flow rate and the smaller the volume of the mixing chamber, the greater will be the sensitivity.

The sensitivity is roughly inversely proportional to the liquid flow rate and directly proportional to the air flow rate. As a practical design limitation the instrument included an air flow rate of from 0.5 to 1.0 l./min. and a liquid flow rate of 42 ml./min. to give a one second sample in the cell. The mixing chamber volume was about 3 ml.

To make the equipment more flexible it was constructed with a multiple switch whereby the load resistor to the photomultiplier could be altered between .1 mg. and 1.0 mg. Other values than those shown may be used and practical limitations are resistors from about 20K to about 5 mg. in these positions.

The total error in the readings made by this equipment is approximately 3 percent and the recorder can be read to one part in a hundred. The sensitivity with organo-phosphorous compounds is of the order of 1 part per million per 0.01 milliamp.

The sampler can also be used for the analysis of chlorine in water or air and of ozone in the atmosphere in connection with air pollution problems.

It is especially effective to check the dissemination of insecticides in the air and to monitor the concentration for safety to livestock and personnel.

The overall response time of the instrument is about 5 seconds. Of this time the actual time required for a sample to be mixed and carried to the cell is about three seconds. The time required to reach maximum intensity is 3 seconds or less. Therefore, the chemiluminescence has attained maximum intensity by the time the mixed sample arrives at the cell.

Since the sampler is not linear over wide ranges of concentration, the instrument should be calibrated with respect to each given compound and set of conditions.

I claim:

A continuous sampler for detecting and measuring the luminescence produced in a luminol solution due to organo-phosphorous compounds in the air comprising:

a transparent cell having a mixing chamber communicating with the lower end of said cell, light sensing means adjacent said cell, said means including a photomultiplier tube, an amplifying circuit

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and a recorder calibrated to measure the amount of said compounds in the air,
an air inlet and a solution inlet connected to said mixing chamber,
a sump connected to said cell for discharging said solution from said cell,
means for equalizing the pressure between said cell and said sump, and
an exhaust connected to said sump for drawing a vacuum through said mixing chamber, said cell and said sump.

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