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TESTING THREE MODELS OF PHOSPHINE DETECTORS

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INTRODUCTION

Work plan in the framework of Sabbatical included demonstration of phosphine gas containment in sealed structures. For this purpose a series of fumigation are planned to be carried out using controlled leak sizes to demonstrate the relationship between the ventilation rate and level of gas containment. This series of studies was termed **"Phosphine Retention (RR)"**. It was necessary to purchase a detector that will cover the range of phosphine concentrations used in commercial fumigation. Three models were received at the HCRL for evaluating their suitability of this particular and future work planned at the same laboratory. Therefore it was decided to test the repeatability, accuracy and working conditions of these instruments.

1- MATERIALS:

A master cylinder containing factory calibrated 5180 ppm of PH3 in N2 atmosphere was used for preparing the different concentrations. This cylinder was equipped with a corrosion resistant stainless steel regulator to reduce gas pressure.

Two floating type rotameters both tested and calibrated for air at 70F and NTP conditions, one model Matheson # 610 with a maximum flow capacity of 100 ml/min, and the other Air Products Brooks # 09-1021 for a maximum flow 1000 ml/min were used for the preparation of the mixtures. The # 09-1021 model rotameter was equipped with two types of floating balls and used simultaneously, one made of glass was used for the lower range and capable of measuring up to 400 ml/min, and the other was of stainless steel to measure up to 1000 ml/min.

The make-up of the mixture to give the desired concentration was obtained from a N2 pressurized cylinder. A pressure resistant 1/4 " external diameter PVC tube was used to connect the pressure lines from the N2 and the PH3 cylinders to the rotameters. In addition a variety of tubes made of Tygon, Latex and PVC was used to make the connection between the rotameters and the supply line to the detectors to be tested.

1.2 **The detectors**

1.2.1.**The ATI PortaSens Gas Detector** - USA

The ATI PortaSens Gas Detector is equipped with an easy pistol grip, a reachargeable NiCad battery, an audible alarm, and a bendable (flexible) metal support probe that facilitates gas sampling. This is an electrolytic type detector. The instrument was designed to display read-out of up to 2000 ppm in intervals of 10 ppm. The internal pump has a suction capacity of 1 SCFH (472 ml/min). It has a removable flow meter to estimate the minimum flow required for taking gas samples. The same instrument may be eqipped with sensors capable of measuring three ranges; the ultra low range capable of measuring the range 0-1000 **ppb** in intervals of 0.1 ppb; the low range measuring 0-10.0 ppm in intervals of 0.1 ppm; and the high range capable of measuring 0-2000 ppm.

1.2.2 - **Drager Miniwarn** Germany:

The tested instrument was Model GD-29-54 Miniwarn – serial number ARMN-1573. This instrument is equipped with two sensors, one capable measuring at the low ranges of 0-10.0 ppm in interavls of 0.01 ppm, the 0-0.2 ppm and the high concentrations measuring in the range of 0-500 in intervals of 1 ppm. It has an audible alarm, a rechargeable battery and computer interface capabilities.

This is a versatile detector capable of measuring different gases including PH3 when equipped with the appropriate electrolytic sensor. It was designed to measure up to 500 ppm in the upper range and 0 to 0.2 ppm at the lower range. The gas samples need to be conveyed to the instrument using an external pump not supplied with the instrument.

1.2.3. - **Bedfont** – England:

This detector has a measuring range of up to 2,000 ppm of PH3. It has a syringe of 60-ml volume connected to one way valve and a T-tube to facilitate gas sampling. It is equipped with two calibration ranges for the zero and the span.

2- METHODS:

Preparation of the gas mixtures was made using the two rotameters in parallel. The PH3 mixture in N2 was further diluted in N2 to obtain target test mixtures of 50, 250, 350, 450, 500, 750, 1000, 1250 and 1500 ppm in N2. The gas mixtures were conveyed through the gas lines shown in Fig. 1.

Fig. 1 – Experimental design of the PH3 and N2 gas lines for obtaining various PH3 concentrations for testing the detectors.

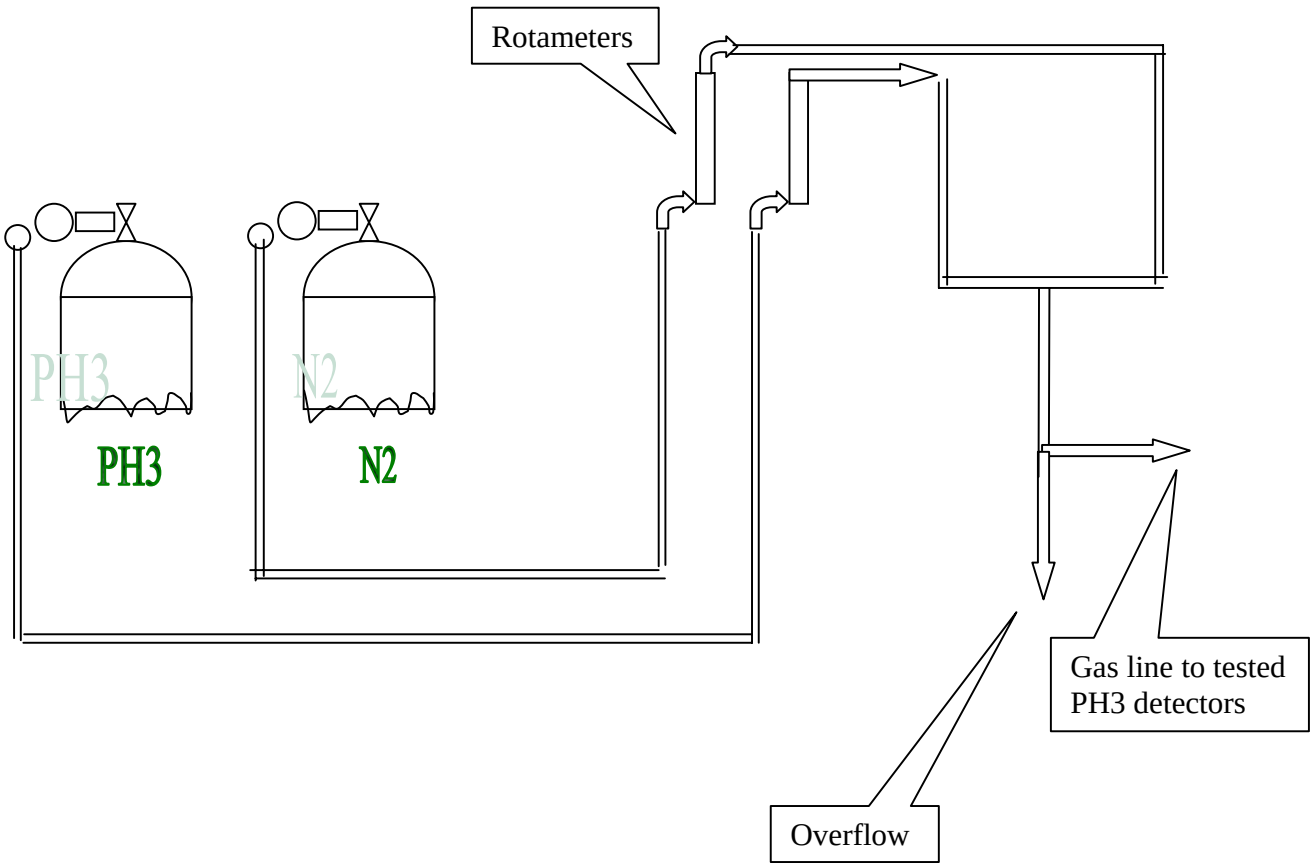
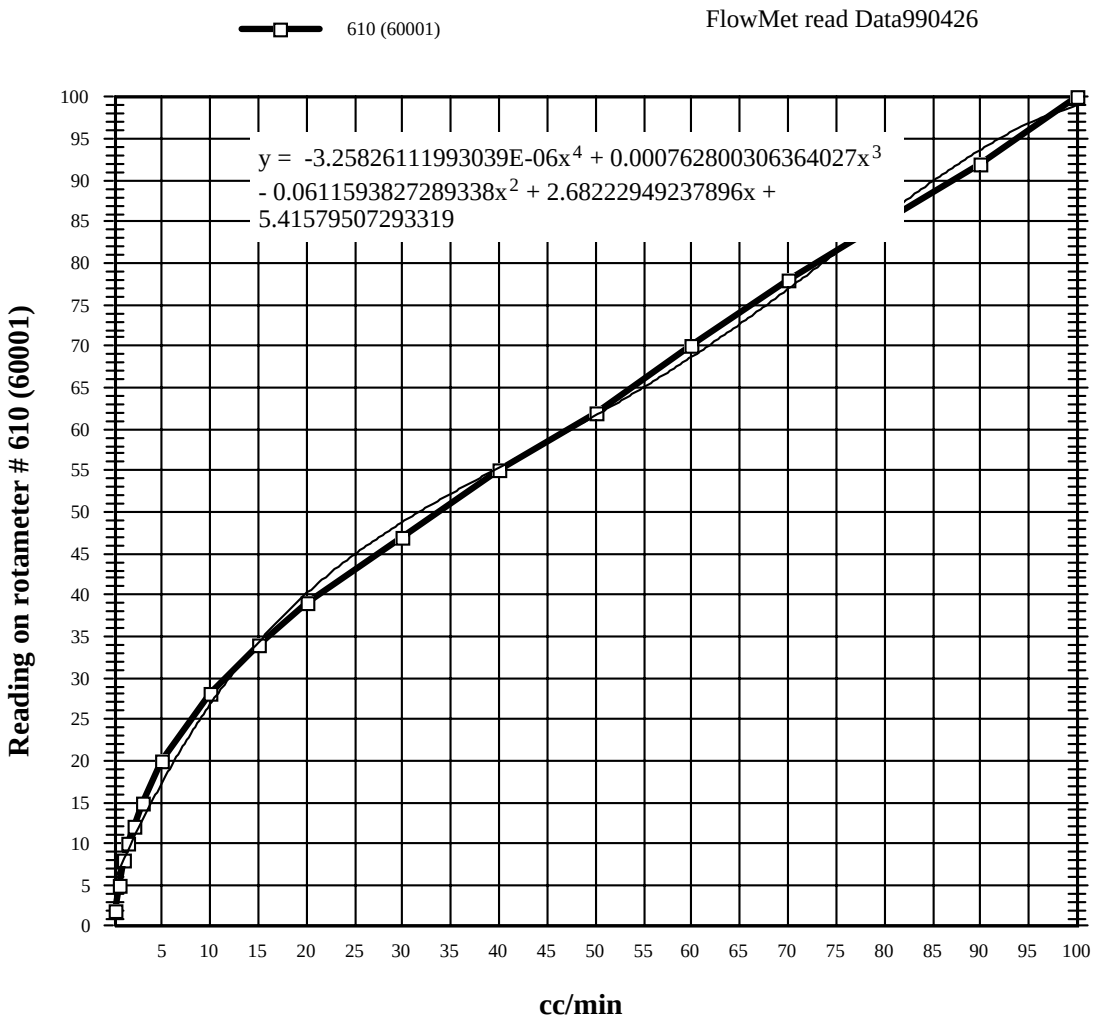


Fig. 2 – Calibration curve and calculated equation to fit the curve for rotameter Matheson # 601, capable of measuring gas flow up to 100 ml/min. This rotameter was used for measuring flow of PH3.



Calibration of rotameters was made by Dave Brandl using air at a temperature of 21^{o}C and 760 mm Hg. Two separate charts were prepared for the rotameters and equations to represent the best fit of these curves are shown in Figures 2 and 3.

The equations obtained from these Figures were used in Table 1 to calculate the set point of the desired gas mixtures.

Fig. 3 – Calibration curves and calculated equations to fit the curve for rotameter Air Products # 09-1021 capable of measuring gas flow up to 1,000 ml/min. This rotameter was used for measuring flow of N2.

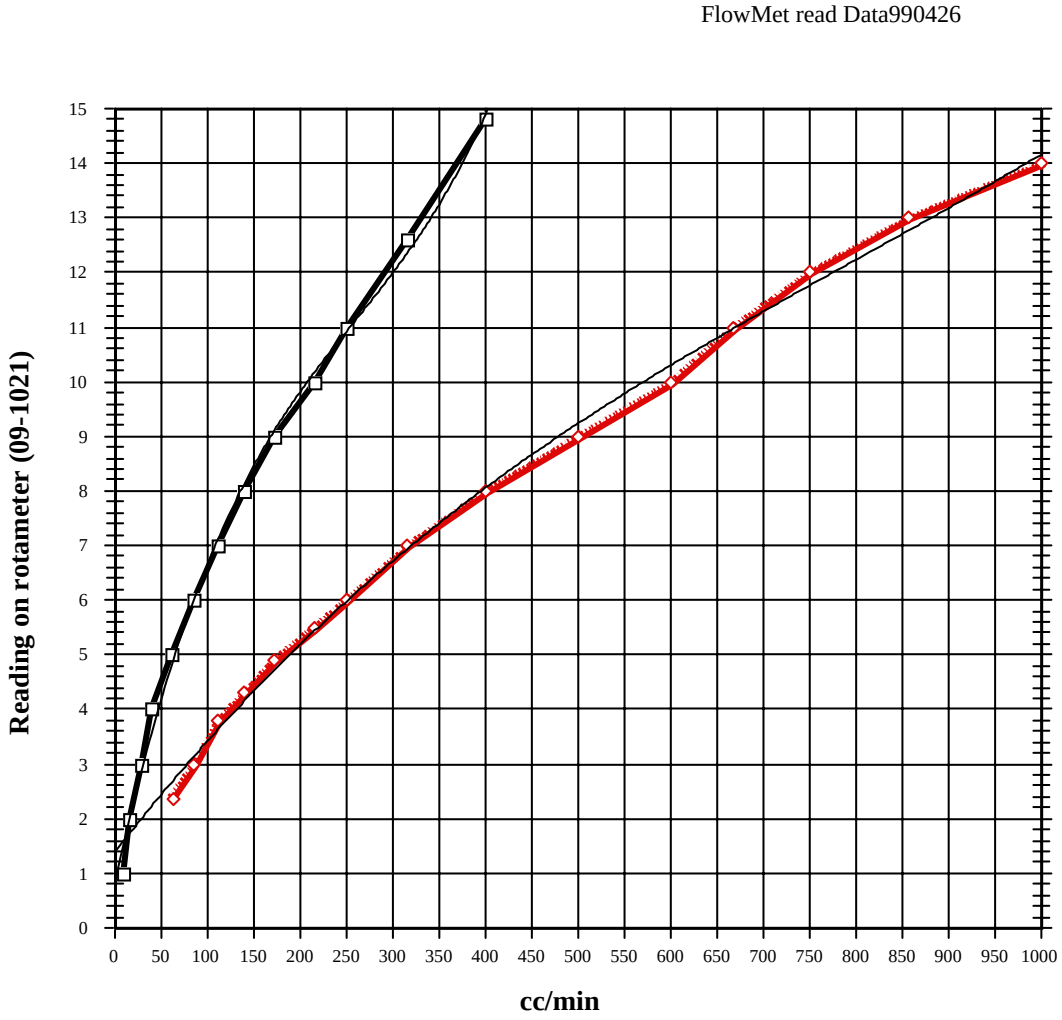


Table 1- Calculated rotameter readings to obtain the desired PH3 concentration in ppm. Table was prepared in an Excel spread sheet to calculate the rotameter readings using the equations representing the curve fits shown in Figures 2 and 3.

Desired PH3 ppm	Total flow ml/min	Initial PH3 ppm in N2	PH3 mix flow ml/min	N2 flow ml/min	PH3 Rot 610 reading	N2 Rot 09-steel	N2 Rot 09-glass
50	100	50	100	0	98.8	1.4	0.8
100	400	5180	8	392	22.8	8.0	14.6
250	400	5180	19	381	39.4	7.8	14.2
300	400	5180	23	377	43.3	7.8	14.1
450	400	5180	35	365	52.0	7.6	13.7
500	400	5180	39	361	54.5	7.6	13.6
750	400	5180	58	342	67.1	7.3	13.0
1000	400	5180	77	323	83.2	7.0	12.5
1250	400	5180	97	303	97.5	6.8	12.1
500	300	5180	87	213	91.3	5.4	10.1
750	250	5180	84	166	89.4	4.6	8.9

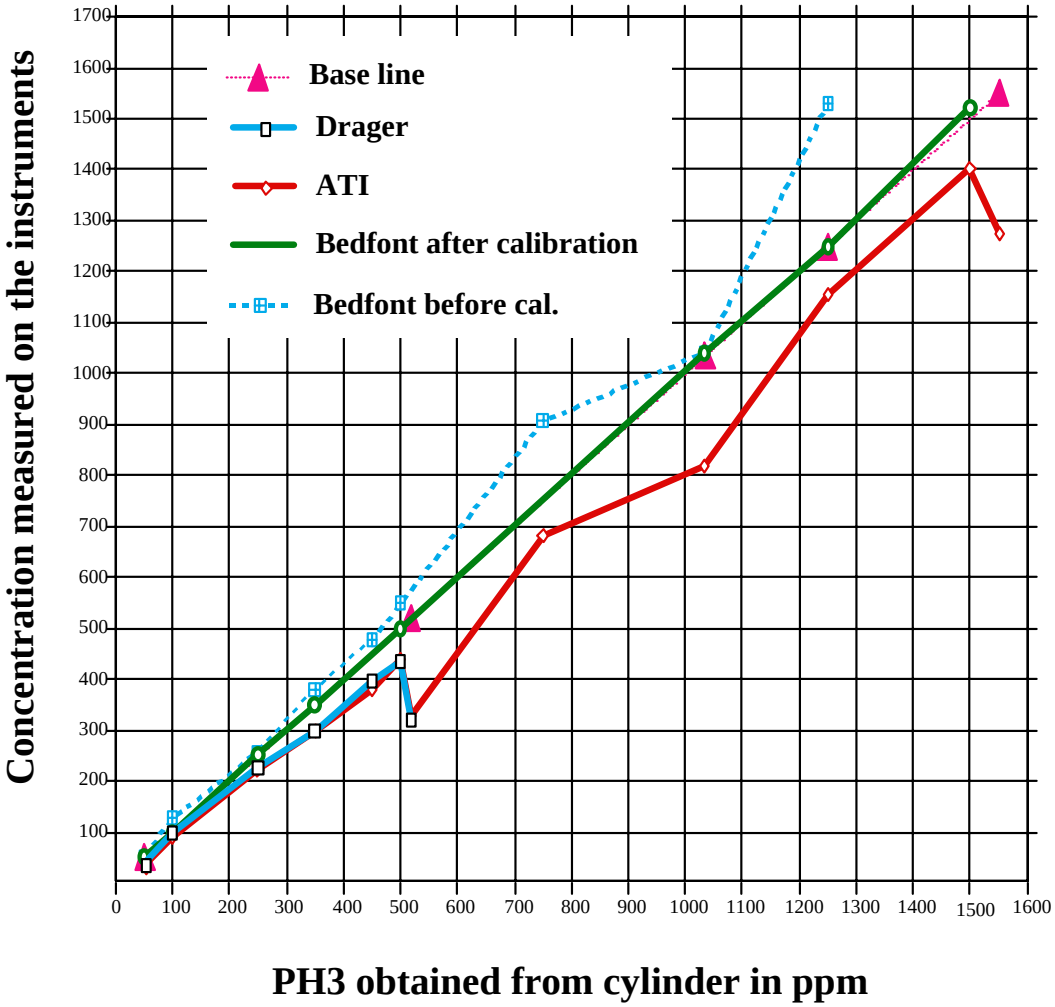
3- **RESULTS**:

Test results were plotted in Fig. 4 showing the measured and calculated PH3 concentrations in N2 atmosphere.

The Drager Miniwarn gave close values at the lower levels of PH3, but as the concentration reached to 500 ppm (maximum measuring capacity), greater deviation from the base line was obtained. It was also difficult to separate the audible alarm deriving from the presence of PH3 in the atmosphere from the battery low signal alarm. The recovery time lasted about 15 min between two readings. Since there was no internal pump, the stream line supplying the PH3 mixture at a rate of 300 to 500 ml/min was used during the tests. The instrument has a strong hysteresis. However, it responds quickly when measuring concentrations higher thn 50 ppm.

The ATI PortaSens Gas Detector measuring 0-2000 ppm in intervals of 10 ppm was tested. Fig. 4 shows that lower readings were obtained compared to the base line. Differences as much as 200 ppm were obtained when tested at concentrations above 500 ppm. The instrument responded very quickly and

Fig. 4 - Comparison of test results obtained with three different instruments. Bedfont was tested before and after calibration.



hysteresis was not apparent. A major disadvantage was that the instrument can be only factory calibrated. Very handy and field-friendly instrument.

Bedfont is equipped with calibration capabilities. Two tests were carried out; before and after calibration. Before calibration the instrument gave close results to the base line at the range of up to 50 ppm. At concentrations higher then 50 ppm there were fluctuations that showed up to 250 ppm deviation from the base line. However, after calibration, it was possible to obtain the closest readings compared to the other two instruments. The sampling using

the syringe was not an easy operation. Therefore, since the instrument is not equipped with an internal pump, the stream line supplying the PH3 mixture at a rate of 300 to 500 ml/min was used during the tests.

4- Conclusions:

- ATI was the only instrument that had an effective internal pump.
- Bedfont was the only instrument that had the calibration capability.
- Drager was capable of measuring up to 500 ppm maximum.
- ATI and Bedfont have the capability of measuring up to 2,000 ppm.
- Closest readings to the base line was obtained using Bedfont after its calibration.
- Drager has a recovery time that extended up to 15 min even when the instrument was esposed to fresh air.
- Bedfont and Drager (at the high range) have the capability to read in intervals of 1 ppm whereas ATI in intervals of 10 ppm.
- In view of the fact that in commercial fumigations the use of concentrations higher than 500 ppm is a common feature, and envisaging that in-situ measuring the PH3 concentrations is the objective for the HCRL, the two instruments ATI and Bedfont were found more appropriate to meet this requirement and therefeore they were recommended for purchase.

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