Procedures for the Generation and Verification of Diisocyanate Atmospheres and the Calibration of Scott Instruments Autostep Plus Monitors

The Scott Autostep Plus Isocyanates Monitor is a direct reading instrument capable of accurately measuring the airborne concentration of 5 different isocyanates. Several users of this instrument have asked for more information on how these various calibration curves were generated. In response to these requests, this Technical Note describes the Autostep Plus calibration process, both during instrument development and during manufacture of finished units.

The heart of the calibration of any air monitoring instrument is the generation of stable test atmospheres of known concentration for the target gas or vapor. This is a trivial matter for gases such as carbon monoxide or methane since they are available in cylinders, as highly accurate mixtures with air. Unfortunately, it becomes a much bigger problem when it is necessary to generate stable atmospheres of reactive materials such as diisocyantes, especially at the the ppb level where Bacharach Autostep Plus instruments are calibrated. An even bigger problem is to accurately know the actual concentration of these reactive gas test atmospheres.

Over the years, Scott has developed procedures for generating and verifying test atmospheres of many reactive compounds. In the case of diisocyantes, the generation method of choice is to allow the compound to diffuse into a stream of nitrogen gas at a constant rate. This rate can be controlled by varying the temperature of the compound and by varying the diameter of the vial used to hold the pure compound. The stream of nitrogen with the diisocyante, typically at 200 cc/min, is fed into a 20 L salinized, glass chamber where it is mixed with a stream of humidified air at 5 - 20 LPM. The concentration of diisocyante is determined by the diffusion rate, the total air and nitrogen flow rate and if any of the diisocyante is reacting with the apparatus.

Because it is well known that materials such as the diisocyanates may react with the walls of the chamber and tubing, it is Bacharach's policy to verify the chamber concentration with analytical procedures that respond only to the reactive diisocyante. Our standard analytical method is to collect an air sample containing the diisocyanates through an impinger containing 1-(2-pyridyl) piperazine (1,2-PP) in acetonitrile. The 1,2-PP reacts with all of the common diisocyantes to form a stable derivative that can be quantitated by HPLC> Standards are prepared by serial dilution of a stock solution of the pure diisocyanate, keeping the 1,3-PP concentration constant. This method has been applied to TDI, MDI, HDI, PPDI, CHMDI and HMDI. We believe that the accurately and precision of this method is on the order of 5% of the actual concentration.

As a routine calibration method, Scott also uses its impregnated tapes as a means of verifying the test atmospheres. A known volume of air is pulled through a piece of tape, selective for the isocyanate. The stain density of this spot is measured on a laboratory reflectance meter and a calibration chart is used to determine the chamber atmosphere concentration. The calibration chart was developed using the atmospheres generated and verified as described above. This technique has proven itself to be rapid and capable of accuracy and precision on the order of 10%. It is this method, which we refer to as a "spot test", that is used in the routine calibration of new and serviced instruments.

Once the procedures and techniques for generating stable and known concentrations of the diisocyanates were developed, the calibration curves for the instrument could be developed. This phase of work starts with the construction of several Autostep Plus instruments and verifying that they pass all of the mechanical and electronic functional tests. Next, the proper impregnated tape is loaded into the instrument and the instrument exposed to at least 5 concentrations of the target isocyanate, verified as described above. We use an external computer to control the test instrument and to collect reflected light levels during these calibration runs. The control of the instrument sampling cycle is important because our instrument calibrations are based on knowing both the reflected light level and the time after the start of a cycle. The "product" of these calibration runs are a series of reflected light versus time (after start of cycle) curves which we call the "family of curves" for a diisocyante.

This family of curves is analyzed and two calibration curves are developed. The first analysis looks at each of the family curves and determines the reflected light level at four minutes after the start of a sampling period. These light levels are "plotted" versus the verified concentration of the target vapor for each curve. This calibration is used for low concentration of the target compound.

The second analysis determines the time it takes the instrument to reach a "threshold" reflected light level when exposed to the known concentrations of the target compound. This time value is "plotted" against the known concentration to yield the second calibration curve. This curve is used by the instrument at high concentrations of the target vapor.

In order the verify the calibration, the instruments are programmed with the new curves and exposed to several more known concentrations of the target diisocyante. The response of the instrument, in concentration units, must agree with the validated concentration to within the stated accuracy specification of the instrument. If this does not happen, the calibration curves are adjusted and the instruments re-tested. This process is repeated until the instrument meets the accuracy specification.

Once this calibration process has been completed, the calibration values are integrated into the instrument firmware used for production units. The design of the Autostep Plus allows the calibrations for up to nine compounds to be stored into the instrument memory. The actual number of calibrations stored in a given instrument is dependent on the intended use of the instrument. These different calibrations in the multiple gas instruments are selected by the range switch on the instrument and by the use of different instrument top caps.

As part of the final test process for every instrument we make, the calibration is verified using two different concentrations of a diisocyante. Typically, TDI vapor, verified by spot test, is used for this validation although, any of the calibrated diisocyanates could be used. If the instrument response falls out or the specified accuracy, then a firmware "span" adjustment is made and the instrument re-tested. Instruments are not released for shipment until they meet the accuracy specifications for that model.

Because of the design of the instrument, it is only necessary to verify the response of each instrument to one of its calibrated compounds. If one compound is within the accuracy specification, then all of the calibrated compounds will be within the specification.