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PART 796—CHEMICAL FATE TESTING GUIDELINES

Subpart A [Reserved]

Subpart B—Physical and Chemical Properties

AUTHORITY: 15 U.S.C. 2603.

Subpart A [Reserved]

Subpart B—Physical and Chemical Properties

§ 796.1050 Absorption in aqueous solution: Ultraviolet/visible spectra.

(a) *Introductory information*—(1) *Guidance information.* (i) Molecular formula.

(ii) Structural formula.

(2) *Standard documents.* The spectrophotometric method is based on national standards and consensus methods which are applied to measure the absorption spectra.

(b) *Method*—(1)(i) *Introduction, purpose, scope, relevance, application and limits of test.* (A) The primary environmental purpose in determining the ultraviolet-visible (UV-VIS) absorption spectrum of a chemical compound is to have some indication of the wavelengths at which the compounds may be susceptible to photochemical degradation. Since photochemical degradation is likely to occur in both the atmosphere and the aquatic environment, spectra appropriate to these media will be informative concerning the need for further persistence testing.

(B) Degradation will depend upon the total energy absorbed in specific wavelength regions. Such energy absorption is characterized by both molar absorption coefficient (molar extinction coefficient) and band width. However, the absence of measurable absorption does not preclude the possibility of photodegradation.

(ii) *Definitions and units.* The *UV-VIS absorption spectrum* of a solution is a function of the concentration, c_i, expressed in mol/L, of all absorbing species present; the path length, d, of the spectrophotometer cell, expressed in cm; and the molar absorption (extinction) coefficient, ε_i, of each species. The

absorbance (optical density) A of the solution is then given by:

$$A = d\sum_i \varepsilon_{ici}$$

For a resolvable absorbance peak, the band width λ is the wavelength range, expressed in nm=10^{-9}m, of the peak at half the absorbance maximum.

(iii) *Reference substances.* (A) The reference substances need not be employed in all cases when investigating a new substance. They are provided primarily so that calibration of the method may be performed from time to time and to offer the chance to compare the results when another method is applied.

(B) Reference compounds appropriate for the calibration of the system are:

(*1*) Potassium dichromate (in 0.005 mol/L, H_2SO_4 solution) from J.A.A. Ketelaar, paragraph (d)(2) of this section:

| log ε .... | 3.56 | 3.63 | 3.16 | 3.50 |
| λ in nm .... | 235 | 257 | 313 | 350 |

(*2*) Fluoranthene (in methanol) from *C.R.C. Atlas of Spectral Data*, paragraph (d)(3) of this section:

| log ε .... | 4.75 | 4.18 | 4.73 | 3.91 | 3.92 |
| λ in nm .... | 237 | 236 | 288 | 339 | 357 |

(*3*) 4-nitrophenol (in methanol) from *C.R.C. Atlas of Spectral Data*, paragraph (d)(3) of this section:

| log ε .... | 3.88 | 4.04 |
| λ in nm .... | 288 | 311 |

See also paragraph (d)(1) of this section.

(iv) *Principle of the test method.* This method utilizes a double-beam spectrophotometer which records only the absorption differences between the blank and test solutions to give the spectrum of the chemical being tested.

(v) *Quality criteria—Reproducibility and sensitivity.* (A) Reproducibility and sensitivity, need not be measured directly. Instead, the accuracy of the system in measuring the spectra of reference compounds will be defined so as to assure appropriate reproducibility and sensitivity. It is preferable to use a recording double-beam spectrophotometer to obtain the UV-VIS spectrum of the test compound. Such an instrument should have a photometric accuracy of ±0.02 units over the absorbance

range of 0 to 2 units. It should be capable of recording absorbances at wavelengths of 200 to 750 nanometers nm with a wavelength accuracy of ±0.5 nm. The cells employed with the instrument must necessarily be transparent over this wavelength range and must have a path length determined to within 1 percent. To ensure that the instrument is performing satisfactorily, spectra for test solutions of $K_2Cr_2O_7$ (for absorbance accuracy) and holmium glass (for wavelength accuracy) should be run periodically.

(B) In the event that a recording double-beam instrument is not available, it will be necessary to determine the absorbance of the test solution in a single-beam instrument at 5-nm intervals over the entire wavelength range and at 1-nm intervals where there are indicated absorbance maxima. Wavelength and absorbance tests should be done as with the double-beam instrument.

(2) *Description of the test procedure—*(i) *Preparation—*(A) *Preparation of test solutions.* (*1*) Solutions should be prepared by accurately weighing an appropriate amount of the purest form of the test substance available. This should be made up in a concentration which will result in at least one absorbance maximum in the range 0.5 to 1.5 units.

(*2*) The absorption of a compound is due to its particular chemical form. It is often the case that different forms are present, depending on whether the medium is acidic, basic, or neutral. Consequently, spectra under all three conditions are required where solubility and concentration allow. Where it is not possible to obtain sufficient concentrations in any of the aqueous media, a suitable organic solvent should be used (methanol preferred).

(*3*) The acid medium should have a pH of less than 2, and the basic medium should be at least pH 10. The solvent for the neutral solution, and for preparing the acidic and basic ones, should be distilled water, transparent to ultraviolet radiation down to 200 nm. If methanol must be used, acidic and basic solutions can be prepared by adding 10 percent by volume of HCl or NaOH in aqueous solution ([HCl], [NaOH]=1 mol/L).

(*4*) In theory, all chemical species other than that being tested are

present in both beams and would therefore not appear in the recorded spectrum of a double-beam instrument. In practice, because the solvent is usually present in great excess, there is a threshold value of wavelength below which it is not possible to record the spectrum of the test chemical. Such a wavelength will be a property of the solvent or of the test medium. In general, distilled water is useful from 200 nm (dissolved ions will often increase this), methanol from 210 nm, hexane from 210 nm, acetonitrile from 215 nm and dichloromethane from 235 nm.

(B) *Blank solutions.* A blank must be prepared which contains the solvent and all chemical species other than the test chemical. The absorption spectrum of this solution should be recorded in a manner identical to that of the test solution and preferably on the same chart. This ''baseline'' spectrum should never record an absorbance reading varying more than ±0.05 from the nominal zero value.

(C) *Cells.* Cell pathlengths are usually between 0.1 cm and 10 cm. Cell lengths should be selected to permit recording of at least one maximum in the absorbance range of 0.5 to 1.5 units. Which set of cells should be used will be governed by the concentration and the absorbance of the test solution as indicated by the Beer-Lambert Law. The cells should be transparent over the range of the spectrum being recorded, and the path-lengths should be known to an accuracy of at least 1 per cent. Cells should be thoroughly cleaned in an appropriate manner (chromic acid is useful for quartz cells) and rinsed several times with the test or blank solutions.

(ii) *Performance of the test.* Both cells to be employed should be rinsed with the blank solution and then filled with same. The instrument should be set to scan at a rate appropriate for the required wavelength resolution and the spectrum of the blank recorded. The sample cell should then be rinsed and filled with the test solution and the scanning repeated, preferably on the same spectrum chart, to display the baseline. The test should be carried out at 25 °C.

(c) *Data and reporting*—(1) *Treatment of results.* (i) The molar absorption coefficient ε should be calculated for all absorbance maxima of the test substance. The formula for this calculation is:

$$\varepsilon = \frac{A}{c_{,i} \times d},$$

where the quantities are as defined above (see Definitions and units).

(ii) For each peak which is capable of being resolved, either as recorded or by extrapolated symmetrical peaks, the bandwidth should be recorded.

(2) *Test report.* (i) The report should contain a copy of each of the three spectra (3 pH conditions). If neither water nor methanol solutions are feasible, there will be only one spectrum. Spectra should include a readable wave-length scale. Each spectrum should be clearly marked with the test conditions.

(ii) For each maximum in each spectrum, the ε value and bandwidth (when applicable) should be calculated and reported, along with the wavelength of the maximum. This should be presented in tabular form.

(iii) The various test conditions should be included, such as scan speed, the name and model of the spectrophotom-eter, the slit width (where available), cell type and path length, the concentrations of the test substance, and the nature and acidity of the solvent medium. A recent test spectrum on appropriate reference materials for photometric and wavelength accuracy should also be submitted (see Reproducibility and sensitivity).

(d) *Literature references.* For additional background information on this test guideline, the following references should be consulted:

(1) Milazzo, G., Caroli, S., Palumbo-Doretti, M., Violante, N., *Analytical Chemistry*, 49: 711 (1977).

(2) Katelaar, J.A.A., *Photoelectric Spectrometry Group Bulletin*, 8, (Cambridge, 1955).

(3) Chemical Rubber Company, *Atlas of Spectral Data*, (Cliffland, Ohio).

[50 FR 39472, Sept. 27, 1985]

§ 796.1950 Vapor pressure.

(a) *Introduction*—(1) *Background and purpose.* (i) Volatilization, the evaporative loss of a chemical, depends upon