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Standard Practice for the Prediction of Contaminant Adsorption On GAC In Aqueous Systems Using Rapid Small-Scale Column Tests[1]

This standard is issued under the fixed designation D6586; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a test method for the evaluation of granular activated carbon (GAC) for the adsorption of soluble pollutants from water. This practice can be used to estimate the operating capacities of virgin and reactivated granular activated carbons. The results obtained from the small-scale column testing can be used to predict the adsorption of target compounds on GAC in a large column or full scale adsorber application.

1.2 This practice can be applied to all types of water including synthetically contaminated water (prepared by spiking high purity water with selected contaminants), potable waters, industrial waste waters, sanitary wastes and effluent waters.

1.3 This practice is useful for the determination of breakthrough curves for specific contaminants in water, the determination of the lengths of the adsorbates mass transfer zones (MTZ) and the prediction of GAC usage rates for larger scale adsorbers.

1.4 The following safety caveat applies to the procedure section, Section 10, of this practice:*This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2652 Terminology Relating to Activated Carbon
D2854 Test Method for Apparent Density of Activated Carbon
D2867 Test Methods for Moisture in Activated Carbon
D2862 Test Method for Particle Size Distribution of Granular Activated Carbon

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms in this practice relating to activated carbon, refer to Terminology D2652.

3.1.2 For definitions of terms in this practice relating to water, refer to Terminology D1129.

4. Summary of Practice

4.1 This practice consists of a method for the rapid determination of breakthrough curves and the prediction of GAC usage rates for the removal of soluble contaminants from water. This is accomplished by passing the contaminated water at a constant controlled rate down flow through a bed of a specially sized granular activated carbon until predetermined levels of breakthrough have occurred.

4.2 When the assumption is made that conditions of constant diffusivity exist within the GAC column, the breakthrough data obtained from the column test can be used to estimate the size and operational conditions for a full-scale carbon adsorber.

5. Significance and Use

5.1 Granular activated carbon (GAC) is commonly used to remove contaminants from water. However if not used properly, GAC can not only be expensive but can at times be ineffective. The development of engineering data for the design of full-scale adsorbers often requires time-consuming and expensive pilot plant studies. This rapid standard practice has been developed to predict adsorption in large-scale adsorbers based upon results from small column testing. In contrast to pilot plant studies, the small-scale column test presented in this practice does not allow for a running evaluation of factors that may affect GAC performance over time. Such factors may

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[1] This practice is under the jurisdiction of ASTM Committee D28 on Activated Carbon and is the direct responsibility of Subcommittee D28.02 on Liquid Phase Evaluation.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

include, for example, an increased removal of target compounds by bacterial colonizing GAC[3] or long term fouling of GAC caused by inorganic compounds or background organic matter[4]. Nevertheless, this practice offers more relevant operational data than isotherm testing without the principal drawbacks of pilot plant studies, namely time and expense; and unlike pilot plant studies, small scale studies can be performed in a laboratory using water sampled from a remote location.

5.2 This practice known as the rapid small-scale column test (RSSCT) uses empty bed contact time (EBCT) and hydraulic loading to describe the adsorption process. Mean carbon particle diameter is used to scale RSSCT results to predict the performance of a full-scale adsorber.

5.3 This practice can be used to compare the effectiveness of different activated carbons for the removal of contaminants from a common water stream.

6. Summary of Practice

6.1 The development of the RSSCT is based on the dispersed-flow pore surface diffusion model (DFPSDM) (Crittenden, et al[5]) which takes into account many of the mechanisms that are known to occur in fixed-bed adsorption. The following mechanisms, which cause the breakthrough curves for an adsorber to spread out and create the mass transfer zone are included in the DFPSDM: external mass-transfer resistance or film transfer, axial mixing due to dispersion and the internal mass-transfer resistances of pore and surface diffusion.

6.2 To simulate full-scale performance, the amount of spreading in the breakthrough curve relative to column depth must be identical for the RSSCT and the full-scale column. To achieve this, the relative contributions of the mechanisms that cause most of the spreading are matched by maintaining similarity as the GAC process is scaled. Studies[5] have shown that matching of the spreading of the breakthrough curve can be achieved by equating the dimensionless groups in PFPSDM (Plug Flow Pore Surface Diffusion Model). Under the conditions that intraparticle diffusivities are assumed to be independent of the carbon particle radius, i.e. the condition of constant diffusivity, the following equation describes the relationship between the small and large columns:

$$\frac{EBCT_{sc}}{EBCT_{lc}} = \left(\frac{R_{sc}}{R_{lc}}\right)^2 = \frac{t_{sc}}{t_{lc}} \tag{1}$$

where: $EBCT_{sc}$ and $EBCT_{lc}$ are the empty-bed contact times for the small-column (RSSCT) and the large-column (full-scale adsorber), respectively; R_{sc} and R_{lc} are the radii of the carbon particles used in the small and large columns, respectively; and t_{sc} and t_{lc} are the elapsed times required to conduct the small- and large-column tests, respectively. The condition of constant diffusivity also requires the Reynolds numbers for the RSSCT and the large-column be equal. This means the following equation must also be satisfied:

$$\frac{V_{sc}}{V_{lc}} = \frac{R_{lc}}{R_{rc}} \tag{2}$$

where: V_{sc} and V_{lc} are the hydraulic loadings in the RSSCT and large columns, respectively. Based upon the above equations, the operating conditions for the RSSCT can be selected to precisely simulate the desired (specified) operating conditions for a full-scale adsorber.

NOTE 1—There is an important issue relating to RSSCT design using Equation 2[6]. Sometimes using leads to a design with a high head loss, which increases dramatically with operating time, as the GAC is crushed by a large pressure drop across the RSSCT. This may be avoided by lowering the superficial velocity as long as dispersion does not become the dominant transport mechanism and intraparticle mass transfer is limiting the adsorption rate. The Peclet number based on diameter can be estimated from the following equation[7]:

$Pe_d = 0.334 \; for \; 160 \leq Re \cdot Sc \leq 40{,}000$

When the velocity is reduced below what is given in Equation A, axial dispersion, which is caused by molecular diffusion, can be more important in the RSSCT than in the full scale process. Consequently, Equation A can be used to check whether dispersion becomes important as the velocity of the RSSCT is reduced in an effort to reduce the head loss. Typical Sc values for SOCs is ~ 2000; consequently, the Re for the RSSCT must be kept greater than ~0.1 and the Pe must be kept above 50 for the length of the mass transfer zone.

NOTE 2—Empty-bed contact time (EBCT) is defined as the bed volume (in liters) divided by the water flow rate in liters/minute. For example if a full scale adsorber holds 20 000 L of activated carbon and the water flow rate is 2500 L/min, the EBCT would be equal to 20 000/2500 or 8.0 min.

6.3 The assumption that conditions of constant diffusivity exist within the GAC column does not apply to all waters or all target compounds. For example this assumption does not apply for the decolorization of water and the adsorption of large molecules, such as humic and fulvic acids. It is recommended that at least one RSSCT pilot-column comparison be conducted to aid in selecting the RSSCT design variables for a given water matrix (Crittenden, et al[5]). A detailed comparison between the constant diffusivity and proportional diffusivity approaches and their respective domains of application is beyond the scope of this practice.

6.4 GAC bed volume and preparation methods are important design parameters for the RSSCT. The GAC bed volume used will determine the required water pumping rate and affect the amount of water needed to complete the test. The minimum column diameter needed to avoid channeling should be 50 particle diameters. For the 10-mm diameter column commonly used in RSSCT systems, a 60 by 80 mesh carbon should be used. Proper GAC sampling (Practice E300) and preparation (grinding, classification and washing) are required for reproducible results.

6.5 Based upon the water feed rate to the column, the time required to reach the desired breakpoint and the weight of

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[3] Owen, D.M., Chowdhury, Z.K., Summers, R.S., Hooper, S.M., and Solarik, G., "Determination of Technology and Costs for GAC Treatment Using the ICR Methodology," AWWA GAC & Membrane Workshop, March 1996, Cincinnati, OH.

[4] Knappe, D., Snoeyink, V., Roche, P., Prados, M. and Bourbigot, M., "The Effect of Preloading on RSSCT Predictions of Atrazine Removal By GAC Adsorbers", *Water Research*, Vol. 31, No. 11, 1997, pp. 2899-2909.

[5] Crittenden, J. C., Berrigan, J. K., Jr., and Hand, D. W., "Design of rapid small-scale adsorption tests for a constant surface diffusivity," *Journal Water Pollution Control Federation*, Vol. 58, No. 4, pp. 312-319, 1986.

[6] Crittenden, J. C., Berrigan, J. K., Jr., Hand, D. W., and Lykins, B. W., Jr. "Design of rapid fixed-bed adsorption tests for non-constant diffusivities," *Journal of Environmental Engineering*, Vol. 113, No. 2, pp. 243-259, 1987.

[7] Fried, J. J., *Groundwater Pollution*. Elsevier Scientific, Amsterdam, The Netherlands, 1975.

carbon used, GAC usage rates for treating the water can be calculated. Breakthrough curves for each contaminant being monitored during the column test can also be generated.

7. Interferences

7.1 Insoluble materials such as oils and greases, suspended solids, and emulsions will interfere with the adsorption of soluble materials by the GAC. Suspended solids in the column feed can lead to increased pressure drop and interfere with the operation of the column. These materials must be removed by suitable means before the water being treated is introduced to the column.

7.2 Air bubbles can interfere with water flow through the column and lead to misleading results. A means for removing air bubbles that are introduced into the system with the feed water should be incorporated to prevent these problems from occurring.

8. RSSCT Test Apparatus

8.1 The RSSCT test apparatus should be constructed of glass, PTFE and/or stainless steel, to minimize the adsorption of organic compounds. The apparatus shown in diagram form in Fig. 1 consists of a metering pump, inlet filter, pressure and flow indicators, up to three columns operating in series and means for water sample collection and analysis.

8.1.1 *Glass columns,* vertically supported, 10.5 ± 0.5 mm inside diameter and approximately 35 cm in length with threaded joints at both ends are most commonly used. Threaded PTFE end caps with seats for neoprene o-ring seals and tubing connectors should be provided at the top and bottom of the column for the admission and discharge of water. For operation at other than room temperature, a means for heating or cooling the columns and the water being treated should be established.

8.1.2 *GAC Support*— A column of fine glass wool installed to give a flat surface across the diameter of the column can be used for support of the GAC column. Alternatively the carbon bed can be supported on a 100-mesh stainless steel screen placed between two short sleeves made from ½ in. PTFE tubing (see Fig. 2). The sleeves should be sized to fit tightly in the column to prevent any fluid from flowing between the sleeves and the column wall.

8.1.3 *Feed Pumps*—A liquid metering pump capable of maintaining a steady flow rate of ± 0.05 mL/min at a column back pressure of up to 100 psig should be used. To prevent over-pressurization of the column system in the event of column plugging during operation, the pump should be set up with a bypass loop that allows the discharge from the pump to be vented back to the pump inlet through an adjustable pressure relief device. The column inlet pressure and water flow rate should be monitored and recorded throughout the run.

8.1.4 *Water Filtration*— A filter to remove suspended solids that may be present in the water should be installed after the metering pump. A 47-mm inline filter housing with a 1.5 µm glass micro-fiber filter has been found to be adequate to remove suspended solids that may prematurely plug the carbon bed. Care must be exercised to ensure organic contaminants in the water being treated are not removed by the filter paper.

8.1.5 *Feed Water Containment*—The feed water should be maintained at the same temperatures as the carbon columns. If the feed water contains volatile organic compounds (VOCs), special care must be taken to prevent their loss during the test. For short duration column tests where a relatively small amount of water is to be treated, the feed water can be stored under zero head space conditions in pillow shaped bags manufactured from PTFE or similar material (typically used for the collection of gas samples). Gas sampling bags up to 100 L in volume can be conveniently used if properly supported. If larger volumes of water containing VOCs are to be treated, a

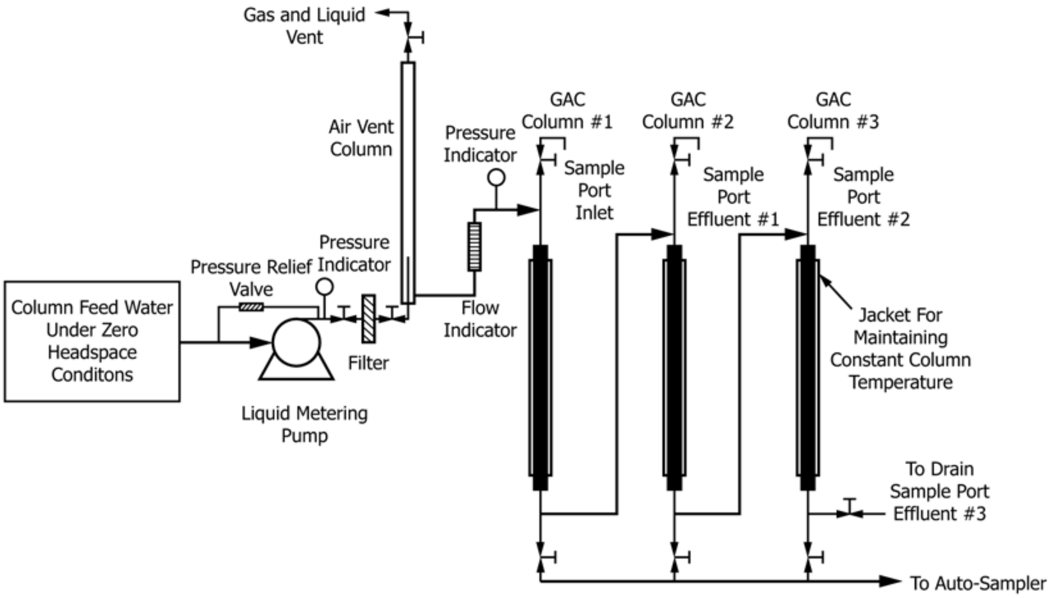

FIG. 1 Flow Diagram for Three Column RSSCT Apparatus

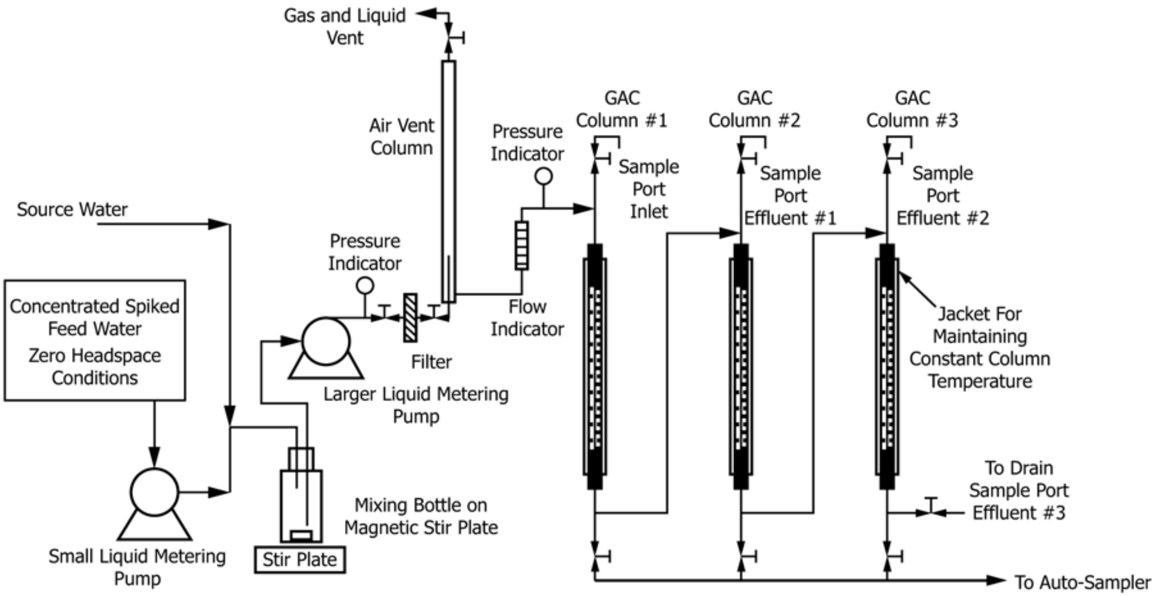

FIG. 2 Alternate Flow Diagram for Three Column RSSCT

55-gal open top drum outfitted with a collapsible PTFE liner or other material that will prevent VOC loss, can be used. The liner is attached to the feed pump inlet tube and collapses as water is removed from the drum, thus always maintaining zero headspace conditions. Jacketed columns with temperature regulated circulation water can be used or the drum can be placed in a temperature-controlled cabinet if control of the feed water temperature is required.

8.1.6 *Sample Collection System*—Water effluent samples for analysis should be collected on a regular basis under zero headspace conditions. The collected samples should be refrigerated during collection or as soon as possible after collection to prevent degradation prior to analysis. The use of an automated sampler that is capable of collecting zero head space samples for extended unattended column tests has been found to be useful for sample collection and preservation. Water flow used in the collection of samples by means of the auto-sampler can be directed to a refrigerated container thus assuring the samples are maintained at cold temperatures during long duration sample collection. The size of the sample collected will be dependent on the type of analysis to be performed. Typically 35 mL vials with PTFE lined screw caps are used for sample collection. Larger water samples (up to 1 or 2 L) can be taken, but due to the low flow rates being used in the column test they will represent the operation of the column over an extended period of time.

8.1.7 *Alternative Column Feed System* —Fig. 3 shows an alternative feed system for the RSSCT. A spiked feed water containing a high concentration of the contaminants to be studied is contained in a zero headspace container as described in 8.1.5. This spiked feed is pumped at a slow rate and mixed with the source water. The flow rates of the two liquid metering pumps are adjusted to give the desired contaminant concentration in the column feed and at the same time the desired total column flow rate to the columns. This setup allows for the easy study of the effect a particular water source matrix on the carbon's ability to adsorb different contaminants at different concentrations.

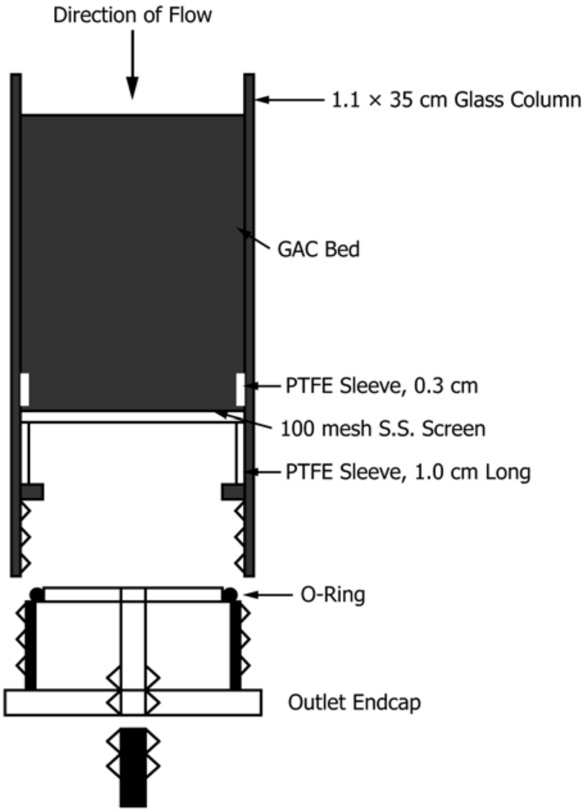

FIG. 3 Diagram of RSSCT Carbon Column

8.1.8 *Sample Analysis*— For column tests of extended duration, the samples collected using the auto-sampler should be analyzed in reverse order, that is, the last sample collected should be analyzed first. If the last sample collected does not indicate breakthrough, the earlier samples collected can be discarded. If breakthrough has occurred, the earlier samples can be analyzed to establish the exact time.

9. Reagents

9.1 *Granular Activated Carbon (GAC).*

9.2 *Water,* ASTM Type IV.

10. Experimental Procedures

10.1 *Carbon Sample Preparations* —A representative sample of the GAC collected according to Test E300 is crushed and screened to yield a 60 × 80-mesh fraction (Test Method D2862). The screened carbon is thoroughly washed with water until all dust is removed and the pH of the wash water shows no significant change. The well-washed carbon is dried at 150°C for 4 h and stored in an amber colored bottle until it is ready for use.

10.2 *Column Loading*— The moisture content (Test Method D2867) and apparent density (Test Method D2854) of the test carbon are determined and recorded. A predetermined quantity of the 60 by 80-mesh carbon is weighed to ± 0.1 mg into a 100-mL beaker (see note). Approximately 40 mL of Type IV water is added to the carbon in the beaker and stirred. The carbon water slurry is heated to boiling for 10 min for the purpose of replacing the air in the carbon's pores with water. After cooling to room temperature, the entire sample of wet deaerated carbon is slurried into the column in such a way as to exclude air bubbles from the column. After loading, the settled height of the carbon column is recorded.

NOTE 3—To determine quantity of carbon needed, first its bed volume is calculated from the EBCT for the column that is required to simulate the full-scale adsorber EBCT (see equation in 6.2) and the water feed rate. Multiplying the bed volume of carbon by its apparent density will give the weight of carbon to be used.

10.3 *Water Feed Rate*— The water feed rate is determined by the empty bed contact time (EBCT) that is required to simulate specified operation in a large-scale adsorber. For example, using the equation from 6.2 and assuming that the $EBCT_{1c}$ is 10 min for the full scale adsorber and $R_{1c} = 0.0792$ cm (the average particle radius for an 8 by 30 mesh carbon) and the particle radius (R_{sc}) for the 60 by 80 mesh carbon used in the column test is 0.0106 cm, the $EBCT_{sc}$ time for the column test would be 0.179 min. The average particle radii for the 8 by 30 mesh and 60 by 80 mesh carbons is determined experimentally by measuring their respective mean particle size (Test Method D2862). In that the volume of the small column (10 mm diameter by 18 cm in depth) to be used is 14.13 cm^3, this example would require a water flow rate of 78.91 mL/min (14.13/0.179) to simulate the full-scale adsorber. For a full-scale adsorber, assuming 10 ft diameter and a water flow rate of 260 gpm, would be 3.3 gpm/ft^2 ($260/(\pi \times 10^2/4)$, or 135 $L/min/m^2$. For the RSSCT the hydraulic loading would be 24.7 gpm/ft^2 ($3.3 \times 0.0792/0.0106$) or 1006 $L/min/m^2$. The column length for the RSSCT would be 18 cm (1006 x 0.179 x 0.1) and the water flow rate required would be 78.91 cc/min if the column diameter is 10 mm ($1006/ (\pi \times 0.01^2/4) \text{ x } 10^{-3}$).

NOTE 4—The average particle radius for the 60 by 80 mesh and 8 by 30 mesh carbons need to be determined experimentally as the magnitude of these values will significantly affect the calculated EBCT. The particle radius can be assumed to be equal to one-half the mean particle diameter as measured using Test Method D2862.

10.4 *Column Operation*— It is often desirable to start the operation of the column(s) using high purity water as the feed. This will allow proper flow rates to be established and ensure there are no air bubbles in the system that will eventually find their way to the carbon column. Once flow rates have been established, column feed is switched to the test water and the auto-sampler (if used) is actuated. The column(s) influent and effluent are sampled and analyzed periodically for the pollutants of interest. The frequency of the sampling will depend on the expected length of the run and the accuracy desired for the determination of the exact breakpoints. The column operation is continued until the effluent analysis equals the influent concentration or until other agreed upon pollutant concentrations are reached. If full breakthrough curves are developed for each contaminate, the length of the mass transfer zone (MTZ) for each compound of interest can be measured. The length of the MTZ can be estimated by first determining the ratio of the time required to go from 5 % breakthrough to 95 % of full breakthrough and the time that has elapsed from the start of the column test to full breakthrough for the compound of interest. Multiplying this ratio by the length of the carbon column measured in centimeters gives the length of the MTZ in centimetres.

10.5 *Data Collection*— The following data is collected during column operation and through subsequent sample analysis and used for the performance predictions:

10.5.1 Carbon bed volume,

10.5.2 Carbon mass used,

10.5.3 Feed water flow rate,

10.5.4 RSSCT EBCT,

10.5.5 Time to specified breakthrough for each contaminate of interest, and

10.5.6 Time to full breakthrough for each contaminate of interest (If Determined).

11. Calculation

11.1 Using data collected from the analysis of the influent and effluent samples and column operating parameters, the following can be calculated:

11.1.1 *RSSCT Bed Life* = Time in minutes to specified breakthrough level

11.1.2 *Treatable Bed Volumes* = RSSCT Bed Life/RSSCT EBCT

11.1.3 *Total Volume of Water Fed to RSSCT Until Breakthrough* = RSSCT bed life by water flow rate,

11.1.4 *Specific Throughput* = Total Volume Fed to RSSCT/ Carbon Mass

11.1.5 *Carbon Usage Rate* = 1/Specific Throughput

11.1.6 *Length of MTZ:*

$$\frac{\text{time to 95 \% BT} - \text{time to 5 \% BT}}{\text{time to Full BT}} \times L \tag{3}$$

where: L = column length.

12. Precision and Bias

12.1 The precision of this practice is limited both by the precision of the standard practices used in determining the concentration of the constituents and the precision of the determination of the exact breakthrough time for each constituent.

12.2 The bias of this practice is limited by the bias of the test methods used in determining the concentration of the constituents and the bias in the determination of the exact breakthrough time for each constituent.

13. Keywords

13.1 activated carbon; column testing; granular activated carbon; RSSCT

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