

Ion Exchange Resin Analysis Guide for the Sweetener Industry

DOWEX<sup>™</sup> Ion Exchange Resin DIRECTOR<sup>SM</sup> Services

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Ion Exchange Resin Analysis Guide for the Sweetener Industry

The Dow Chemical Company offers ion exchange resin testing services to its customers for a nominal fee. This guide provides information to help you get the most from Dow's DIRECTOR<sup>SM</sup> Services offering. It provides helpful instructions for obtaining your resin sample, labeling it, mailing the sample, and contacting Dow. It also provides forms to help us collect the information which will allow us to provide you with the most useful recommendations for troubleshooting or optimizing your process.

DOWEX<sup>™</sup> Ion Exchange Resins and DOWEX OPTIPORE<sup>™</sup> Adsorbents can be tested to determine suitability for continued operations or to troubleshoot problems in your resin system. In addition to test results, you will receive a complete report with a description of the tests performed and the implications of the test results on your operation.



The following information will help you initiate the resin testing process.

# Sampling and shipping procedures

In order to obtain representative results, all resin samples sent for testing must be suitably representative of the resin in the entire bed. One option is to take a core sample from the resin bed. Another option is to take separate samples from multiple locations from representative areas within the bed and combine them. And yet another option is to sample from all locations in a moving slurry of resin, such as can be obtained during a backwash. If a sample thought to be representative of the entire bed of resin is not available, please indicate on the label the location in the bed from which the sample was obtained. The samples should be shipped in tightly sealed, leak-free one-liter plastic bottles placed in plastic bags for secondary leak containment. MAKE SURE THE SAMPLES DO NOT CONTAIN FREE CAUSTIC OR ACID. The solutions should be at neutral pH for safe shipping.

Labeling is critical. Bottles should be labeled with company and plant names, contact name and phone number, resin name, and bed identification. Also indicate whether the resin is regenerated or exhausted. Additionally, the enclosed Analysis Information Sheet should be completed and submitted with resin samples so test results can be properly interpreted.

After packaging, the resins should be shipped to:

The Dow Chemical Company Larkin Laboratory Attn: Ion Exchange Lab #124 1801 Larkin Center Drive Midland, MI 48674 USA

# Information needed to begin testing

#### Application information

In order to begin testing, Dow laboratory personnel need to have proper information on the types of resin being sent and the types of testing to be performed. The resin names and descriptions on sample bottle labels identify the types of resins submitted and your completed System Information Sheet will identify your application and typical conditions. Standard tests for each type of resin will be performed as listed in the next section.

# Standard Testing Services Available

The following test options are available, based on the application of the resin. Tests are performed on each resin component. For example, mixed bed resins are separated and tested as two separate resin components.

#### Strong Acid Cation Resins

Water Retention Capacity (WRC) Total Exchange Capacity (TEC) Microscopic Bead Examination

#### Weak Base Anion Resins

Water Retention Capacity (WRC) Total Exchange Capacity (TEC) Weak Base Capacity (WBC) Salt Splitting Capacity (SSC) Weak Base Dynamic Test (Op-Cap\* & Rinse\*) Microscopic Bead Examination

#### **Mixed Bed Resins**

Standard testing for Strong Acid Cations Standard testing for Strong Base Anions Cation/Anion Resin Ratio

#### Chromatographic Separation Resins

Water Retention Capacity (WRC) Total Exchange Capacity (TEC) Microscopic Bead Examination Metal ionic forms of sites, percent

#### Strong Base Anion Resins

Water Retention Capacity (WRC) Total Exchange Capacity (TEC) Weak Base Capacity (WBC) Salt Splitting Capacity (SSC) Microscopic Bead Examination

#### **Adsorbent Resins**

Water Retention Capacity (WRC) Microscopic Bead Examination Organic Foulant Extraction and UV Classification

\* Op-Cap is a high flowrate water treatment test which checks WBA resins for fouling; rinse volume is one component of this test. Additional tests are available based on your specific needs. See below.

# Tests Available for an Additional Fee

Water Retention Capacity as received (WRC as received) Total Exchange Capacity as received (TEC as received) Particle Size Distribution

## **Communication and Billing information**

In addition to the resin and system information, a purchase order number or credit card information is required before testing will begin. This information should be provided with the shipment to expedite the process. Pricing can be obtained from your Dow sales representative or this <u>price sheet</u>. A valuable option to simplify the process for you is to pre-purchase services in advance. This simplifies and expedites the paperwork and payment process. Consult your Dow sales representative and your company's purchasing contact if this is of interest for you.

Provide at least one contact name with telephone number, and E-mail address with your samples so laboratory personnel can obtain any additional or missing information required to conduct the analysis.

# Availability of results

Results are generally available within 4 weeks after your samples, complete resin application information, and complete contact person and billing information are received at our laboratory site. Rush testing is available for an additional fee.

## **Contact your Dow representative**

If you have any questions regarding ion exchange resin testing, contact your Dow representative. If you cannot identify your Dow Representative, please call 1-800-447-4369 (North America), +800-3-694-6367 (Europe) or +800 7776 7776 (Pacific) for assistance, or see the *Contact Us* section of our website at <u>http://www.dowwaterandprocess.com</u>.

# Available Testing Services For Ion Exchange Resins for the Sweetener Industry

The Dow Chemical Company offers ion exchange resin analysis services for a nominal fee. The following analyses are available for the sweetener industry:

## Water Retention Capacity (WRC)

Measurement of the inherent moisture content of the ion exchange resin. A fully hydrated resin sample is centrifuged to remove free water, and the resulting sample is weighed before and after drying to calculate the water content as a percent of the original hydrated resin weight. Elevated water content indicates degradation of the polymer chain, while depressed water content typically results from accumulation of foulants on the resin beads. This test is performed on cation resins while in the H<sup>+</sup> form, strong base anion resins while in the Cl<sup>-</sup> form and weak base anion resins while in the free base form.

#### Total Exchange Capacity (TEC)

Measurement of the total number of ion exchange sites per volume of resin. Fully regenerated resin is titrated to obtain milliequivalents of capacity per milliliter of resin. For cation resins, this property is reported in the H<sup>+</sup> form. For strong base anion resins, it is reported in the Cl<sup>-</sup> form. The TEC of weak base anion resins are reported in the free base form.

#### Weak Base Capacity (WBC)

Measurement of the number of weak base (tertiary amine) sites available on an anion resin. For weak base anion resins, the WBC is the critical parameter indicating the ability of the resin to remove acids. For strong base anion resins, the WBC is negligible initially but may increase as the salt splitting sites are converted to weak base sites during use. This property is reported for weak base anion resins in the free base form and for strong base anion resins in the CI<sup>-</sup> form.

## Salt-Splitting Capacity (SSC)

Measurement of the strong base (quaternary amine) sites on the resin. For weak base anion resins, the SSC is a minor component of the capacity; with time SSC is lost due to fouling of the strong base sites. It is important for controlling the swell of the resin, but a SSC that is too high can cause syrup degradation products. For strong base anion resins, the SSC is the major component of the resin capacity. It is these sites which act in concert with the strong acid sites of the cation resin to perform mixed bed polishing. This property is reported for strong base anion resins in the CI<sup>-</sup> form and for weak base anion resins in the free base form.

## Weak Base Dynamic Test (Op-Cap)

This test is used to measure organic fouling of weak base anion resins by examining resin kinetic performance. It yields two numbers, rinse volume and operating capacity. The test can be critical to understanding resin performance since the resins are used dynamically in the plant. The results can be independent from results under static conditions and are important to a total understanding of resin state. Rinse volume is the amount of water required to rinse the resin down to a conductivity of 50  $\mu$ S/cm after caustic regeneration. Operating capacity (op-cap) is an indication of the level of organic fouling of the resin. It measures the ion exchange sites used under dynamic conditions that, in our test, stress the kinetics beyond normal operating conditions.

#### Microscopic Bead Examination

A sample of the resin is placed under a microscope for determination of the physical integrity of the resin and contamination of the bed. Photographs are taken and included with the report.

#### Percent Capacity in Specific Ionic Forms

This test can be performed on any cation resin, but is primarily useful for chromatographic separation resins. A separation resin must be in the desired ionic form for maximum performance. This test analyzes the amount of specific ions on the resin by stripping the metals from the regeneration with acid followed by atomic absorption of the stripped solution and reports the percentage of the sites in the ionic forms. Metal analyses available include:  $Ca^{2+}$ ,  $Mg^{2+}$ ,  $K^+$ ,  $Na^+$ , and  $Fe^{2+}$  or  $Fe^{3+}$ .

## **Additional Tests**

#### Water Retention Capacity as received (WRC as received)

This is a measurement of the water content of the resin before laboratory regeneration. This is generally measured on a plant-regenerated resin to evaluate the efficiency of regeneration achieved in your process. The weight of the resin is measured before and after drying to calculate the internal water content of the resin. This can be compared against the results of a laboratory-regenerated sample of the same resin sample.

#### Total Exchange Capacity as received (TEC as received)

This is a measurement of the number of ion exchange sites in the regenerated ( $H^+$  or  $OH^-$ ) form as regenerated in your process. This result can be compared against the total exchange capacity after laboratory regeneration to determine the efficiency of regeneration.

#### Particle Size Distribution

A sample of the resin is tested with an instrument which has been calibrated to determine the particle size distribution of ion exchange resins. The particle size distribution is reported along with a number of particle size distribution parameters.

# Resin Analysis Services for the Sweetener Industry - see separate price sheet

## Service notes

- Standard services require approximately 4 weeks to complete from the time of receipt of resin samples and purchase order number or credit card information at Dow. Data can be made available within 2 weeks of receipt of resins and purchase order or credit card information for an additional rush analysis fee. The formal report would follow within the standard 4 week time frame. Special requests will vary; please consult your Dow Chemical representative for cost estimates and timing.
- Each of the services above includes written documentation indicating the results and recommendations. A written report will be sent by E-mail to E-mail addresses provided on the original paperwork. Also, a hard copy of the written report is mailed/faxed to one location of the client's choice.
- All service items are net 30 days.
- Dow's standard terms and conditions apply.
- Customers exclusive remedy for all claims (INCLUDING BREACH OF WARRANTY, NEGLIGENCE AND STRICT LIABILITY) related to these services is limited to a refund of the price paid for the service which is the subject of the claim. DOW shall NOT be liable for consequential, incidental, punitive, special or exemplary damages.

# **DOWEX™** Ion Exchange Resins

Analysis Request Form

#### **Customer Information**

Send Results To:		Send Invoic	e To:			
Name:		Name:				
Company:		Company:				
Plant Name:						
Address:		Address:				
Phone:		Phone:				
Fax:		Fax:				
E-mail:		E-mail:				
Purchase Order Number:						
Credit Card Number:	Expiration Date:		Select Credit Card Typ	ce: Card American Express		
(The credit card option is available only Note: For all Analysis Request	in the USA and Canada) s originating outside the	USA and Canada, a P	P.O. number is required.			
Rush Analysis Requested: (Additional Fees Will Apply) List specific samples to be analyzed on a rush basis:						
Sample Descriptions:						
Train ex: Dextrose, Fructose	Vessel ex: 1, 2, A, C, etc.	Resin Type ex: Strong acid cation	Ex: DOWEX™ 88 SAC resin	Special Tests		

Contact names and addresses, purchase order number or credit card information, and sample descriptions must be complete before testing will be initiated. (Note: Analysis results may be delayed if this information is not provided.) Standard analysis and report will be available within four weeks. Please complete this form and mail it, along with the properly packaged resin samples, to the address below. For questions, please call 1-800-447-4369.

The Dow Chemical Company Larkin Laboratory Attn: Ion Exchange Lab #124 1801 Larkin Center Drive Midland, MI 48674 USA



# **Technical Information**

Feed Composition Analysis:

Dextrose Side	MB Polisher	Adsorbent
Fructose Side	Separation	
Corn Syrup	Other:	

Persistent or recurring problems:

Vessel for Sample No.	1	2	3	4	5	6
Identification						
Diameter (ft)						
Height of Resin Bed (ft)						
Height of Vessel (ft)						
Resin Type						
Lot Number(s), if possible						
Approx. Date Installed (Mo/Yr)						
Has it Been Topped Off?						
Approx. Rebed Time, months						
Regenerant Used and Concentration						
Regeneration Temperature Range						
Regenerant Flowrate						
Regenerant Volume						
Co- or Counter-current Regeneration Mode?						
Cross-Regeneration Frequency						
Cross-Regenerant Used and Concentration						

# **DOWEX™ Ion Exchange Resins**

Resin Sampling Procedure

The utility of this device is to obtain a representative sample of a resin bed from the top of the bed to the bottom with minimum equipment expense.

## Step No. 1

Build the device as shown. It is important that the lower stopper be rounded or it will not seat properly. A rubber ball may be substituted in place of the stopper, but its diameter must be larger than the pipe's diameter.

Other Required Equipment:

- 2-3 gallon (~10-liter) clean plastic bucket
- 1-quart (~1-liter) plastic sample jars
- Plastic storage bags for secondary leak containment

## Step No. 2

Open access to the resin vessel. This is a good time also to measure the resin depth and inspect the upper distributors. It is not always necessary to remove the manway. If there is a nozzle larger than 2 inches (5 cm), it can be used for this procedure.

#### Step No. 3

Drain the excess water in the vessel to resin level. Make sure that no free acid or caustic is present. If necessary, rinse the resin to ensure the resins are at neutral pH.

#### Step No. 4

Allow the lower stopper to extend 6 inches (15 cm) from the bottom of the PVC pipe. Use the upper stopper to hold the string in this position.

#### Step No. 5

Using a slow up and down motion, insert the device into the resin bed slowly. It may help to rotate the pipe as it is moving down into the bed of resin. This must be done slowly to allow the resin level to equalize in the pipe. Inserting the device too fast will give you a sample of only the bottom portion of the bed.

#### Step No. 6

When the device hits bottom, pull it back 2-3 inches (5-8 cm) and pull the string to seat the lower stopper. Pushing down on the pipe will aid in seating the stopper. Stretch the string tight and insert the upper stopper to hold it.

#### Step No. 7

Remove the device from the bed and lower the bottom end of the pipe to a person on the floor. Remove the upper stopper. Remove the lower stopper and allow the resin to discharge into the bucket.

#### Step No. 8

Pour deionized water in the top of the pipe to rinse the resin out of the pipe. Repeat the procedure as many times as required to obtain 1 quart (-1 liter) of resin. Pour collected resin into 1-quart (-1-liter) plastic sample bottles. Seal the sample bottles with tape and place in plastic storage bags for secondary leak containment prior to packaging.

#### Step No. 9

Proper labeling is essential. If resins are to be shipped for testing, label the bottle with company name, plant name, contact name and phone number, resin name, and bed identification and number. Also indicate whether the sample is regenerated or exhausted.

#### Step No. 10

Send the resin samples to your resin testing supplier. Remember to include system information as well as which tests should be performed on each resin. For questions about Dow's resin testing services, please contact your Dow representative and ask about DIRECTOR<sup>SM</sup> Services.

WARNING: The resin will come out with a lot of force in some cases. Use caution and wear eye protection.



# **DOWEX™** Ion Exchange Resins

Shipping Instructions Form

This shipping information is provided for your convenience as part of DIRECTOR<sup>SM</sup> Services. It contains instructions for sampling, labeling and mailing ion exchange resin samples for analysis.

The Dow Chemical Company offers ion exchange resin testing services to its customers and potential customers for a nominal fee. Ion exchange resins can be tested to determine suitability for continued operation or to troubleshoot problems in your resin system. In addition to test results, you will receive a complete report with a description of the test performed and the implications of the test results for your operation.

#### Sampling and shipping procedures

In order to obtain representative results, a representative sample is required. This is critical. One way to attempt to obtain a representative sample is to take a core sample from the resin bed. The samples should be shipped in 1-liter (1-quart) plastic bottles placed in plastic bags for secondary leak containment. MAKE SURE THE SAMPLES DO NOT CONTAIN FREE CAUSTIC OR ACID AND THAT NO GLASS CONTAINER IS USED. The solutions should be at neutral pH for safe shipping.

Labeling is critical. Bottles should be labeled as shown in the example below.

Resin Name	
Resin Type	
Vessel / Line Number	
Resin sampled after regeneration	Yes – No
Date sample was taken	

#### Warnings for handling ion exchange resin:

- Avoid contact with eye
- In case of contact, rinse with plenty of water and seek medical advice
- Resins in regenerated form may leach acid or basic solution when in contact with water
- Wash hands after sampling
- Ship resin in neutral solution
- Ship in plastic bottle
- Package each bottle inside a sealable plastic bag

Contact names and addresses, purchase order number (or credit card information) and sample descriptions (given on the Analysis Request Form) must be completed before testing will be initiated. Any technical information you can provide on this form is important so that test results can be properly interpreted. A standard analysis and report will be available within 4 weeks of Dow receiving the resin sample and documentation.

# Contact Dow Water & Process Solutions:

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http://www.dowwaterandprocess.com

Notice: Oxidizing agents such as nitric acid attack organic ion exchange resins under certain conditions. This could lead to anything from slight resin degradation to a violent exothermic reaction (explosion). Before using strong oxidizing agents, consult sources knowledgeable in handling such materials.

Notice: No freedom from infringement of any patent owned by Dow or others is to be inferred. Because use conditions and applicable laws may differ from one location to another and may change with time, Customer is responsible for determining whether products and the information in this document are appropriate for Customer's use and for ensuring that Customer's workplace and disposal practices are in compliance with applicable laws and other governmental enactments. The product shown in this literature may not be available for sale and/or available in all geographies where Dow is represented. The claims made may not have been approved for use in all countries. Dow assumes no obligation or liability for the information in this document. References to "Dow" or the "Company" mean the Dow legal entity selling the products to Customer unless otherwise expressly noted. NO WARRANTIES ARE GIVEN; ALL IMPLIED WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE ARE EXPRESSLY EXCLUDED.

