This article was downloaded by:
On: *25 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK

Separation Science and Technology

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

The Efficiency of the Dynamic Tests Used to Evaluate the Adsorbents Used for Separation of C_8 Isomers

Miroslaw Milewski[a]; Elzbieta Brzostowska[a]; JÓZef M. Berak[a]
[a] INSTYTUT CHEMII PRZEMYSŁOWEJ, WARSAW, POLAND

To cite this Article Milewski, Miroslaw , Brzostowska, Elzbieta and Berak, JÓZef M.(1983) 'The Efficiency of the Dynamic Tests Used to Evaluate the Adsorbents Used for Separation of C_8 Isomers', Separation Science and Technology, 18: 2, 187 — 193

To link to this Article: DOI: 10.1080/01496398308055668

URL: http://dx.doi.org/10.1080/01496398308055668

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

SEPARATION SCIENCE AND TECHNOLOGY, 18(2), 187–193, 1983

NOTE

The Efficiency of the Dynamic Tests Used to Evaluate the Adsorbents Used for Separation of C_8 Isomers

---

MIROSŁAW MILEWSKI, ELŻBIETA BRZOSTOWSKA,
and JÓZEF M. BERAK

INSTYTUT CHEMII PRZEMYSŁOWEJ
RYDYGIERA 8, 01-793 WARSAW, POLAND

Downloaded At: 13:36 25 January 2011

Abstract

Results of liquid chromatographic tests used to screen zeolitic adsorbents for resolving xylene isomer mixtures are presented. The performances of two tests (pulse test and breakthrough test) were studied. The selectivities and standard deviations obtained, showing that the pulse test is less sensitive, are presented.

INTRODUCTION

The interest in the use of adsorptive techniques for separating and purifying organic congeners, amplified in the 1960s by the advent of processes for making highly efficient zeolite adsorbents, has given strong impetus to the development of new testing techniques in which the actual process conditions are adequately reproduced. Reasons for this tendency are quite obvious—investment risk and prestige jeopardized if an unsuitable adsorbent is put on the market are rather high. Hence the companies leading in the design of continuous adsorption processes are among those actively developing new testing procedures.

This also holds true for processes used to separate xylene isomers. The present version, involving simulation of the adsorbent bed movement, was introduced by the Universal Oil Products Company in the 1970s (*1*). In numerous references the UOP has also described the methods for selecting adsorbents and the techniques for testing the quality of adsorbents (*2, 3*), e.g., Ref. *4*, published in this Journal, was concerned with UOP's dynamic tests.

Copyright © 1983 by Marcel Dekker, Inc.

0149-6395/83/1802-0187$3.50/0

Downloaded At: 13:36 25 January 2011

In this Institute practical application studies have been conducted for several years, preceded by basic research concerning the process for isolating *p*-xylene from C_8 isomers and terminated by the development of selective adsorbents (*5*, *6*), a mathematical model of the process (*7*), and pilot plant studies (*8*). Adsorbent quality problems were also included.

It is the purpose of the present study to check, with the above-mentioned process in mind, the reproducibility and efficiency of UOP's dynamic tests reported for relatively low-selective adsorbents (separation coefficient $\sim$3).

CHARACTERISTICS OF THE DYNAMIC TESTS

The dynamic tests available for column chromatography are of two types: column breakdown and pulse. The former test is continued until the column breaks down; the test involves contacting the adsorbent with the feed (a mixture to be separated) until the mass transfer process is completed, i.e., the concentrations at the inlet and outlet of a column are identical, and the components taken up are desorbed.

Elution curves are analyzed and the selectivity of the adsorbent is evaluated. The desorbate usually contains a small proportion of a nonadsorbing component, the presence of which allows the dead volume of the column to be evaluated (in full analogy to gas chromatography). The principle of the test is evident from Fig. 1 in which the method for evaluating the selectivity is also presented.

The pulse test employs the principle which has long been used to determine the adsorptive properties of sorbents applied in gas mixture separation processes. The test involves saturating the adsorbent with a desorbent, feeding a feed pulse, and after desorbent dosing has been restored, recording the variation of the composition with time. The principle of the test and a method for evaluating the adsorbent quality are given in Fig. 2.

EXPERIMENTAL

In the present investigation the zeolite used was a BaKNaY zeolite granulated into spheres 0.4–0.8 mm in size with a composition warranting its low selectivity.

The testing apparatus (Fig. 3), analogous to UOP's, consisted of a thermostated column packed with the adsorbent, a pressure control system, feedstock supply, desorbent supply, and analysis systems. A Chromatron (GDR) model GChF-18.3 gas chromatograph was used for analysis. It was

Downloaded At: 13:36 25 January 2011

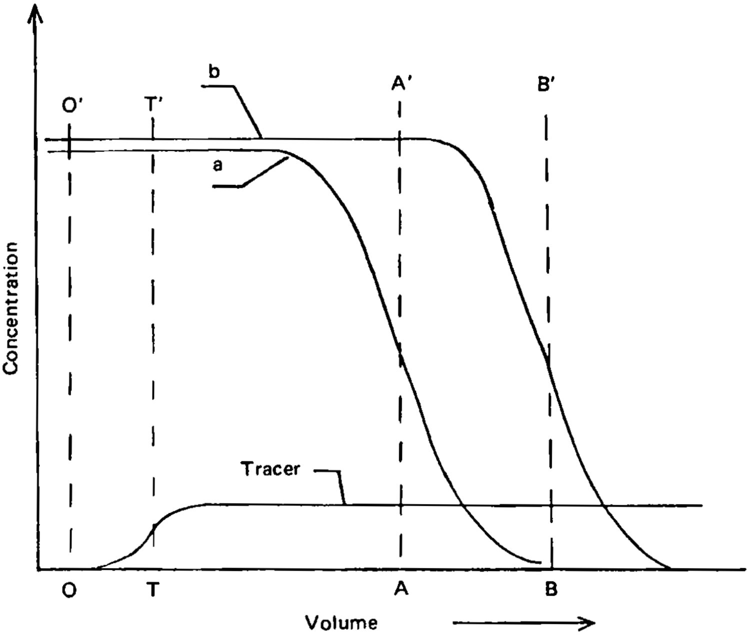

FIG. 1. Column void volume = OT. Capacity for $a = TT'A'A = C_a$. Capacity for $b = TT'B'B = C_b$. Selectivity $(b/a) = C_b/C_a$.

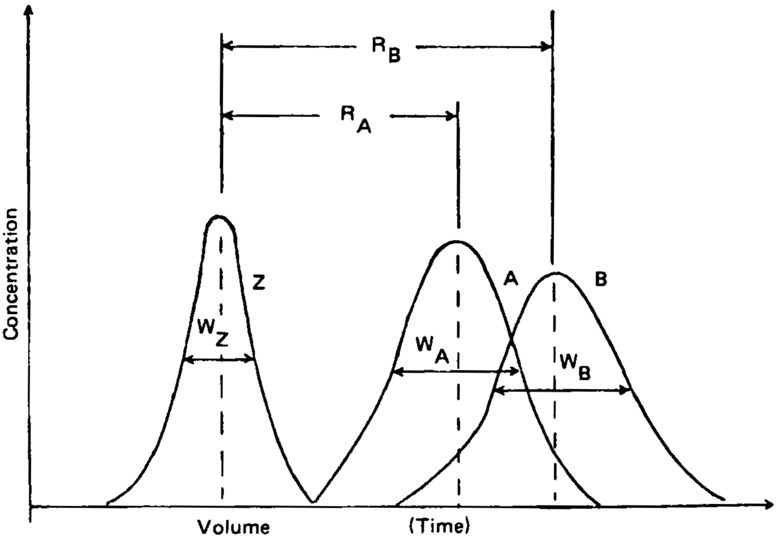

FIG. 2. R_A = net retention volume of A. R_B = net retention volume of B. Selectivity $(B/A) = R_B/R_A$. W_z, W_A, W_B = envelope half-widths A, B, Z.

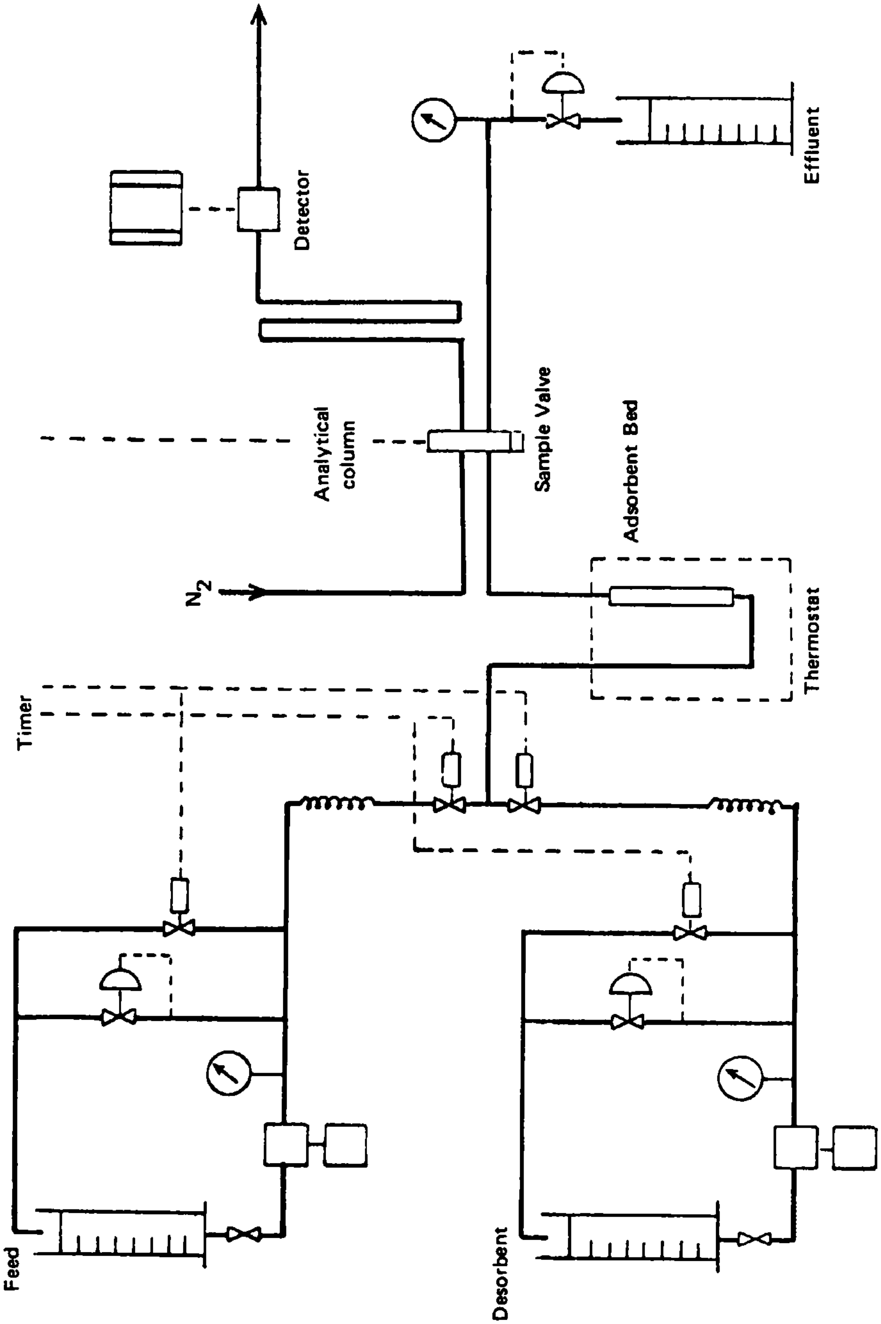

FIGURE 3.

Downloaded At: 13:36 25 January 2011

equipped with a 2-m long Chromosorb WAW DMCS/1.5% di-*n*-nonyl phthalate + 1.5% bentone 34 column. The column and the injection chamber were kept at temperatures of 353 and 473 K, respectively.

The tests continued until the breakdown point of the column was reached. They were carried out with a feedstock composed of isooctane 75, ethylbenzene 6.25, *m*-xylene 6.25, *o*-xylene 6.25, and *p*-xylene 6.25 vol%. The desorbent contained isooctane 73, *n*-octane 2, and toluene 25 vol%. The desorbent and the adsorbent were supplied by means of a nonpulsating pump to a 1200 × 10-mm column at a rate of 1 mL/min. The column was maintained at a temperature of 423 K and a pressure of 0.7 MPa.

RESULTS AND DISCUSSION

Selectivity of the adsorbent was characterized in terms of the separation coefficient evaluated from the experimental data according to the procedures presented in Figs. 1 and 2. Results of the determinations are summarized in Tables 1 and 2.

Table 1 presents the results of selectivity determinations obtained in the test continued until the breakdown point of the column was reached. As is evident from Table 1, the least-scattered data are those obtained with the isomers closely related in sorptive capability (*p*-xylene and ethylbenzene). As the sorptive capabilities diverge, the error of the determination increases owing to the fact that diverse-sorbed components produce elution curves following disparate curves and thus the maxima in the curves are less precisely determined.

Downloaded At: 13:36 25 January 2011

TABLE 1
Breakthrough Test Results

Experiment	Selectivity[a]		
	$B_{p/m}$	$B_{p/\mathrm{etB}}$	$B_{p/o}$
1	1.76	1.16	1.67
2	2.40	1.44	2.39
3	1.93	1.25	1.71
4	2.56	1.27	1.89
5	1.61	1.21	1.50
6	1.99	1.08	1.65
Mean value	2.04	1.80	1.23
Standard deviation	0.368	0.314	0.121

[a] *p*, *m*, *o*, and etB are *para-*, *metha-*, *ortho*-xylenes, and ethylbenzene, respectively.

TABLE 2
Pulse Test Results

Experiment	Selectivity		
	$B_{p/m}$	$B_{p/\mathrm{etB}}$	$B_{p/o}$
1	2.52	1.25	2.52
2	2.57	1.29	2.57
3	2.08	1.30	2.08
4	2.00	1.18	2.00
5	2.70	1.60	2.70
6	2.04	1.45	2.04
7	2.34	1.40	2.34
8	2.40	1.44	2.40
Mean value	2.31	1.36	2.31
Standard deviation	0.25	0.18	0.26

Downloaded At: 13:36 25 January 2011

Table 2 summarizes the pulse test data. The separation coefficients for *p*-xylene relative to those for the *meta* and *ortho* isomers are identical (maxima in the elution curves for these isomers overlap). The former test revealed no such coincidence. Therefore, the tests were checked for correlation by using a modified statistic procedure (*6*) to establish whether or not the results of the tests differ significantly. The check showed the results of the tests to correspond with each other with a small error. Therefore, to determine the selectivity of adsorbents, each test may be used alternatively, except that UOP's original pulse test is the least sensitive. For industrial practice, this is of negligible importance because *m*- and *o*-xylene are received as a single stream (raffinate), hence the minor differences in their sorption capabilities need not be determined and knowledge of these data are important only for basic research work.

To improve the precision of the dynamic test, we suggest phase-shifted tests be carried out. Preliminary results indicated a considerable increase in the precision of the test with only a minor protraction ($\sim$20%) of the experimental time. The procedure applied, the apparatus, and the results obtained will be reported in detail in a forthcoming paper.

REFERENCES

1. U.S. Patent 3,960,774 (1976).

2. A. J. de Rosset, R. W. Neuzil, D. J. Korons, and D. H. Rosback, *Separation of C_8 Aromatics by Adsorption,* Presented at ACS Meeting, California, March-April 1971.

3. A. J. de Rosset, R. W. Neuzil, and D. J. Korons, *Liquid Column Chromatography as a Predictive Tool for Continuous Countercurrent Adsorptive Separation,* Presented at 169th Meeting of ACS, Pennsylvania, April 1975.

4. A. J. de Rosset, R. W. Neuzil, D. J. Tojbl, and J. M. Braband, *Sep. Sci. Technol., 15*(3), 637 (1980).
5. M. Milewski and J. M. Berak, 5th French-Polish Catalysis Congress, Toruń, 1975.
6. Polish Patent 105,904 (1979).
7. M. Milewski, J. M. Berak, and R. Kazimierczuk, *Przem. Chem., 58,* 531 (1979).
8. J. Berak and M. Milewski, 1st Polish-Italian Symposium on Selected Problem in Catalysis, Warsaw, 1979.

Received by editor July 27, 1982

Downloaded At: 13:36 25 January 2011