Dist. Category UC-45

HNS ASSAY BY LIQUID CHROMATOGRAPHY

C. L. Schaffer

DEVELOPMENT DIVISION

JULY - SEPTEMBER 1977

Process Development
Endeavor No. 107

MASTER

Mason & Hanger-Silas Mason Co., Inc.
Parlex Plant

P. O. BOX 30020
AMARILLO, TEXAS 79177
806-335-1581

operated for the
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
under
U. S. GOVERNMENT Contract EY 76 C 04 0487

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately-owned rights.

Printed in the United States of America
Available from
National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161
Price: Printed Copy \$4.00; Microfiche \$3.00

HNS ASSAY BY LIQUID CHROMATOGRAPHY

C. L. Schaffer

DEVELOPMENT DIVISION

Process Development
Endeavor No. 107

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

ABSTRACT

An improved method for assay of hexanitrostilbene (HNS) by liquid chromatography was developed using a μ Bondapak NH_2 column.

DISCUSSION

The existing process for analytical analysis of HNS utilizes liquid chromatography with three columns (1 m x 2.1 mm interdiameter), packed with Corasil II. A carrier solution of 20% dioxane and 80% heptane, with a flow of 1.2 ml per minute is employed. HNS is injected using dioxane as the solvent with a concentration of 0.50 mg HNS per ml. Fig. 1 shows a typical chromatogram using Corasil II.

An improved method has been developed using a Waters Associates μ Bondapak NH_2 packed column. This column has a high polarity, strongly basic surface functionality, created by chemically bonding a monomolecular layer of an aminopropylsilane to a small particle, full porous support. This packing material is normally used for difficult analytical and semi-preparative separations of strongly polar compounds. The same solvent system was used, except the percentages of dioxane and heptane were changed. This procedure was developed based on experiments with many different column packings and carrier solvents. All carrier solvents were compatible with the 254 nm UV detector used.

Fig. 1. Chromatogram of HNS
Sample by Corasil II

The μ Bondapak NH_2 column was 30 cm in length by 4 mm interdiameter. The carrier solvent was 28% dioxane and 72% heptane. The injection solvent and concentration remained the same as used for the Corasil II columns. A typical μ Bondapak NH_2 chromatogram of HNS is shown in Fig. 2. This system gives better resolution and efficiency than the previously used Corasil II, as can be seen by comparing Figs. 1 and 2. The capacity of the μ Bondapak column is also greater than the Corasil II, which permits a larger amount of HNS be injected into the column if needed, which can improve both the quantitative and qualitative analyses.

The injection method was changed from a syringe to a micro-sampling valve. Repeatable and precise sample injections are more easily achieved with a sample valve injection.

CONCLUSIONS

Quantitative analysis of HNS was developed using a μ Bondapak NH_2 column with a pure HNS sample of known concentration and a prepared sample of HNS with known quantities of impurities added. Comparing the HNS band areas from the chromatograms, an experimental error of $\pm 1/2\%$ was observed for the quantitative analysis of the HNS.

Fig. 2. Chromatogram of HNS Sample by μ Bondapak NH_2

DISTRIBUTION

ALO

V. C. Vespe, Director
Weapons Development Division

R. R. Fredlund, Jr., Director
Classification & Technical
Information

Mound Lab

Director of Explosives

SLL

C. S. Selvage - Org. 8180

LLL

G. L. Dittman (10 Copies)
Attn: R. J. Woodworth

C. T. Brockett, Technical Information
Dept.

LASL

E. H. Eyster - WX-DO
R. N. Rogers, Attn: F. B. Baker - WX-2
J. Aragon - WX-3
Report Library - ISD-4

SLA

B. E. Arthur - Org. 1570
J. C. Crawford - Org. 2500/D. H. Anderson -
Org. 2510
J. C. King - Org. 2300/C. B. McCampbell
Org. 2310
Central Technical Files - Org. 3141

UK

Jack Kirkham
Atomic Weapons Research Establishment
Aldermaston, Reading, England

PX

Division Manager, Mfg. Engineering
Division Manager, Quality
Division Manager, Development (2 copies)
B. H. Carr, E&TC
Technical Library

Circulation Copy:

- (1) Area Manager, DOE/AAO
- (2) Division Manager, Manufacturing
M&H

Circulation Copy:

- (1) C. R. Poole
- (2) Plant Manager
- (3) M&H-SM Co., Inc., Lexington, KY