

AR226 - 3260

DuPont Haskell Laboratory for Health
and Environmental Sciences

November 4, 2002

cc: MR- [REDACTED]
Quality Assurance

TO: MR FILE [REDACTED] POCKET H-25425

FROM: J. C. Maslanka JCM

TEST SUBSTANCE STABILITY OF H-25425

Medical Research Project Number:
Haskell Sample Number:
Haskell Reference Standard Number:
Analytical Test Code:
Analytical Report Number:

[REDACTED]
25425
25425
[REDACTED]
HA-2002-094

Notebook References:

[REDACTED]

Attached is the analytical report for the study identified above. This analysis was performed to satisfy protocol requirements for test substance stability but may also be used for other purposes.

TEST SUBSTANCE STABILITY OF H-25425

Medical Research Project Number:
Haskell Sample Number:
Analytical Report Number:


25425
HA-2002-094

A sample of H-25425 was submitted on October 14, 2002 and analyzed on October 15, 2002. The percentage of active ingredient (a.i.) was measured to be  with a range of  of nominal for replicate analyses (n = 3). The sponsor reported a purity of  when the sample was submitted.

ACKOWLEGEMENTS

Sample preparation by Sheila A. Riley (Chemistry Associate). Sample analysis by Bogdan Szostek (Senior Research Chemist).

SIGNATURE

Report by:


Janet C. Maslanka
Analytical Staff Chemist

4-Nov-2002
Date

Date Issued:

4-Nov-2002

METHODS

Analysis for the percent of a.i. in a sample of H-25425 was by high-performance liquid chromatography (HPLC) with a Gel Permeation Column (GPC) and refractive index detector according to the following method. Sonication was used to completely dissolve the test substance and the analytical standard stock solution.

SAMPLE PREPARATION & ANALYSIS

Aliquots (0.0413, 0.0407 and 0.0412 grams) of H-25425 were dissolved in tetrahydrofuran (10 mL) to give nominal concentrations of 4130, 4070, and 4120 ppm, respectively.

CHROMATOGRAPHIC CONDITIONS

Instrument:	Hewlett-Packard Model 1100 HPLC
Column:	Styragel® HR 4; 7.8 mm x 300 mm (GPC column)
Mobile Phase	100% tetrahydrofuran (THF)
Flow Rate:	1.0 mL/min
Injection Volume:	50 µl
Column Temperature:	35°C
Detection:	Refractive Index

CALIBRATION & QUANTITATION

A separate sample of H-25425 test substance was designated as the analytical reference standard (93.17% pure). A stock solution of H-25425 in tetrahydrofuran was used to make calibration solutions that bracketed the test solutions. Peak heights from replicate HPLC/GPC/RI analysis of each calibration solution were used to construct a calibration curve by least-squares regression. Measured concentrations for each test solution were determined by applying respective peak heights to the calibration curve.

RESULTS

H-25425 eluted from the HPLC column as a resolved peak with a retention time of about 8.582 ± 0.01 minutes. The sponsor reported a purity of [REDACTED] when the sample was submitted.

Table I. The percent of a.i. in the H-25425 sample analyzed October 15, 2002.

Aliquot	ppm H-25425		Percent Nominal
	Nominal	Measured	
1	4130	3730	90.3
2	4070	3720	91.4
3	4120	3790	92.0
Average % Nominal			91.2
Standard Deviation			± 0.85
Coefficient of Variation			1%