DuPont Haskell Laboratory for Health
and Environmental Sciences

March 6, 2003

cc: MR [redacted]
Quality Assurance

TO: MR FILE [redacted] POCKET H-25425

FROM: J. C. Maslanka

TEST SUBSTANCE STABILITY OF H-25425

Medical Research Project Number:	[redacted]
Haskell Sample Number:	25425
Haskell Reference Standard Number:	25425
Analytical Test Code:	[redacted]
Analytical Report Number:	HA-2003-032
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: [redacted]
Haskell Sample Number: 25425
Analytical Report Number: HA-2003-032

A sample of H-25425 was submitted on February 26, 2003 and analyzed on February 27, 2003. The percentage of active ingredient (a.i.) was measured to be [redacted] with a range of [redacted] of nominal for replicate analyses (n = 3). The sponsor reported a purity of [redacted] 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 6-Mar-2003
Janet C. Maslanka — Date
Analytical Staff Chemist

Date Issued: 6-Mar-2003

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 (0.0403, 0.0417 and 0.0409 grams) of H-25425 were dissolved in tetrahydrofuran (10 mL) to give nominal concentrations of 4030, 4170, and 4090 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 9.023 ± 0.02 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 February 27, 2003.

	ppm H-25425		Percent
Aliquot	Nominal	Measured	Nominal
1	4030	3730	92.6
2	4170	3790	90.9
3	4090	3780	92.4
Average % Nominal			**92.0**
Standard Deviation			**± 0.9**
Coefficient of Variation			**1%**