

Terms & Conditions

Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not otherwise permitted to reproduce, republish, redistribute, or sell any Supporting Information from the ACS website, either in whole or in part, in either machine-readable form or any other form without permission from the American Chemical Society. For permission to reproduce, republish and redistribute this material, requesters must process their own requests via the RightsLink permission system. Information about how to use the RightsLink permission system can be found at <http://pubs.acs.org/page/copyright/permissions.html>



ACS Publications

MOST TRUSTED. MOST CITED. MOST READ.

Copyright © 1997 American Chemical Society

Chiral HPLC Conditions for:

(*S*)-4-Ethyl-3,4,6,10(4*H*)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1*H*-pyrano-[3,4-*f*]-indolizine (1)

Chiralcel OD column (4.6 mm x 25 cm)

Mobile phase: 65:35 hexane/EtOH + 0.1% TFA

Flow rate: 1 mL/min

Detector: 335 nm

Injection size 10 µL

Retention times:

(*S*)-1, 13.2 min

(*R*)-1, 16.2 min.

Sample preparation

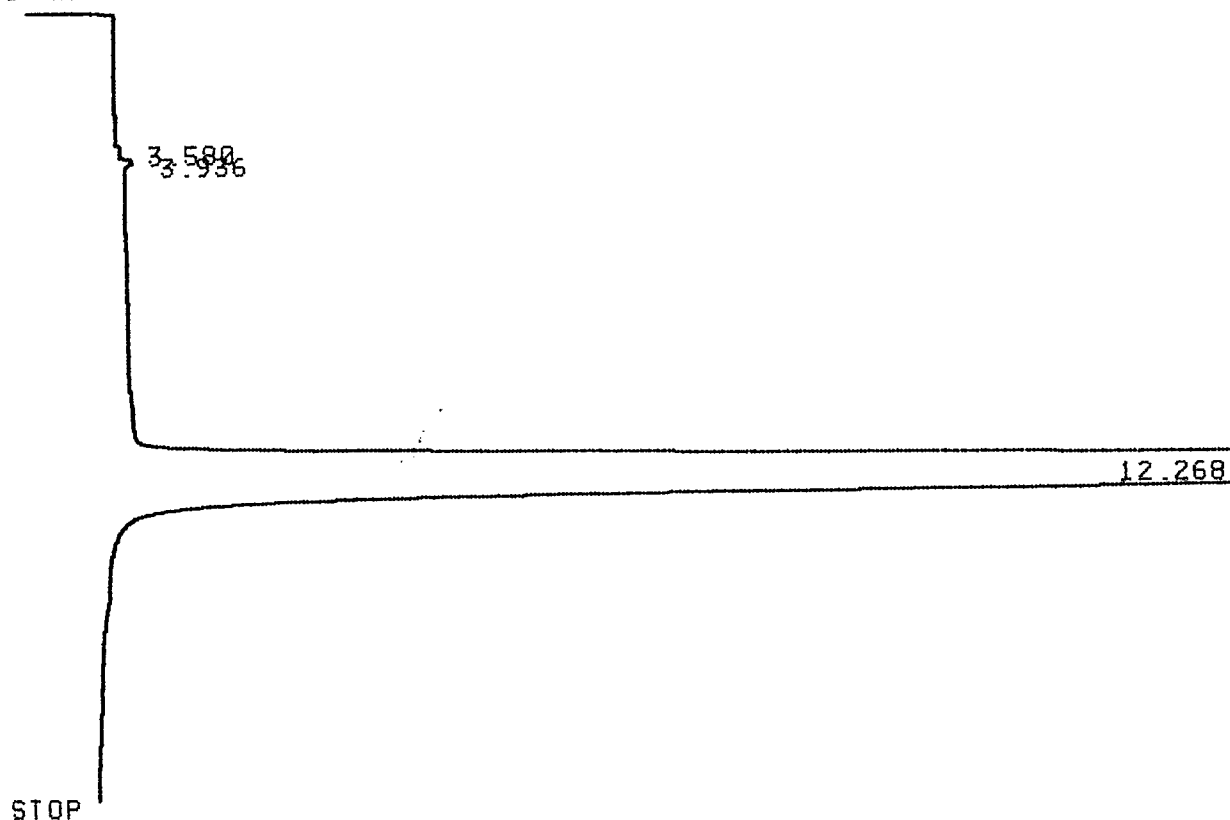
0.01g compound **1** dissolved in 5 mL ethanol and then diluted with 15 mL Mobile phase.

EE determination was made by dissolving 0.2502g compound **1** in 125 mL ethanol and diluting to a volume of 500 mL with the mobile phase. HPLC trace labelled 1 is a direct injection of this solution. The sample for HPLC trace 2 has 0.1% of *R*-1 added (1.0 mL of a solution of 0.0251g *R*-1 (ee >98%) dissolved in 100 mL ethanol). The sample for HPLC trace 3 has 0.2% *R*-1 added. HPLC trace 4 is racemic compound **1**.

HPLC 1

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)

* RUN # 1529 JUN 12, 1997 09:08:56
START



RUN# 1529 JUN 12, 1997 09:08:56

RT	AREA	TYPE	WIDTH	AREA%
3.580	10502	BU	.121	.02256
3.936	46066	UU	.200	.09898
12.268	46484960	UB	.646	99.87846

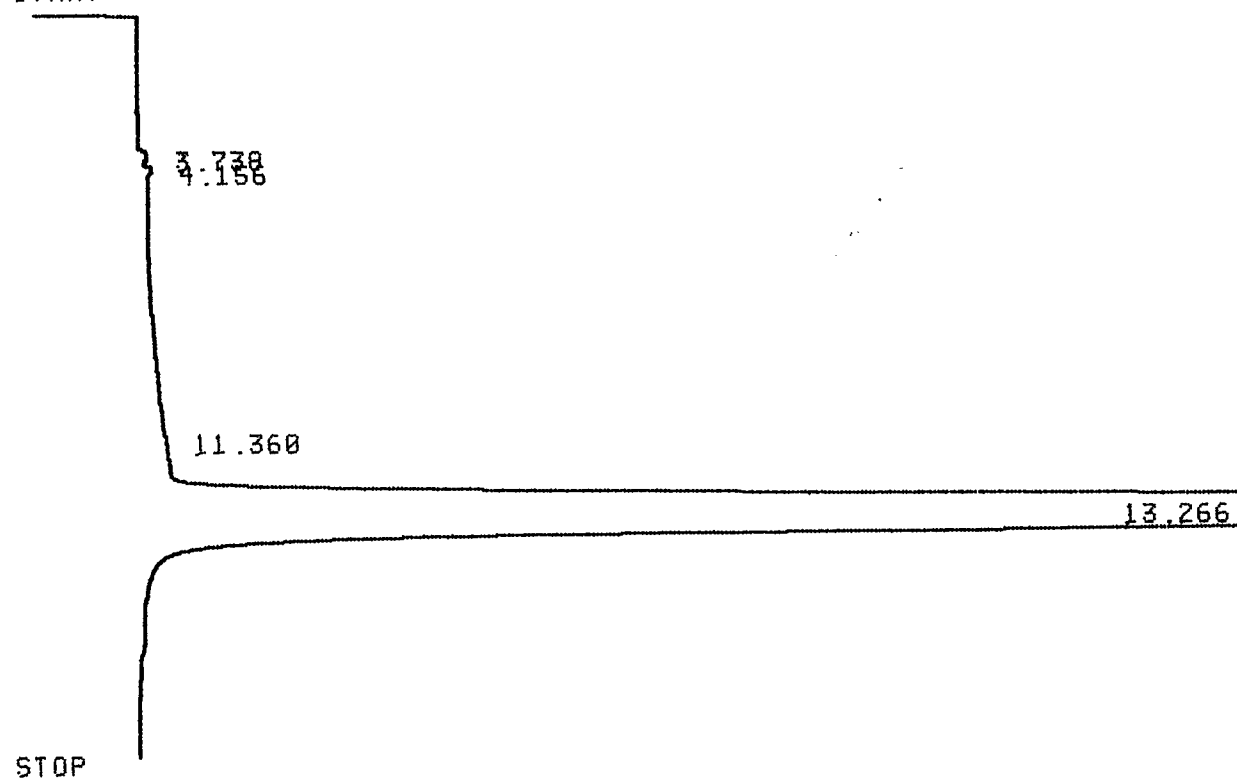
TOTAL AREA=4.6542E+07
MUL FACTOR=1.0000E+00

HPLC 2

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-
[3,4-f]-indolizine (1) + 0.1% R Enantiomer

* RUN # 1530 JUN 12, 1997 09:47:11

START



STOP

RUN# 1530 JUN 12, 1997 09:47:11

AREA%

RT	AREA	TYPE	WIDTH	AREA%
3.738	14361	PV	.124	.03190
4.156	27102	UB	.193	.06020
11.360	131093	BU	.086	.29121
13.266	44843936	UB	.637	99.61670

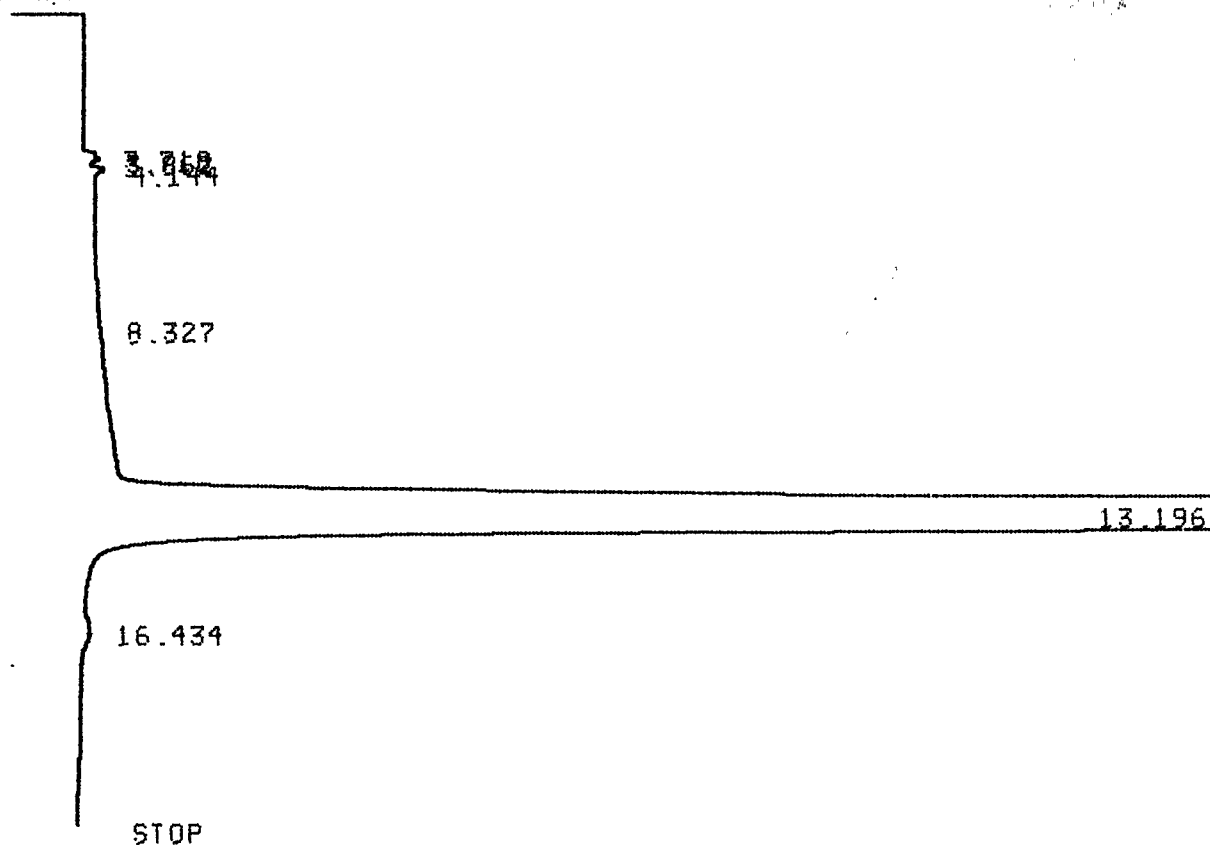
TOTAL AREA=4.5016E+07

MUL FACTOR=1.0000E+00

HPLC 3

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1) + 0.2% R Enantiomer

* RUN # 1531 JUN 12, 1997 10:07:28
START



RUN# 1531 JUN 12, 1997 10:07:28

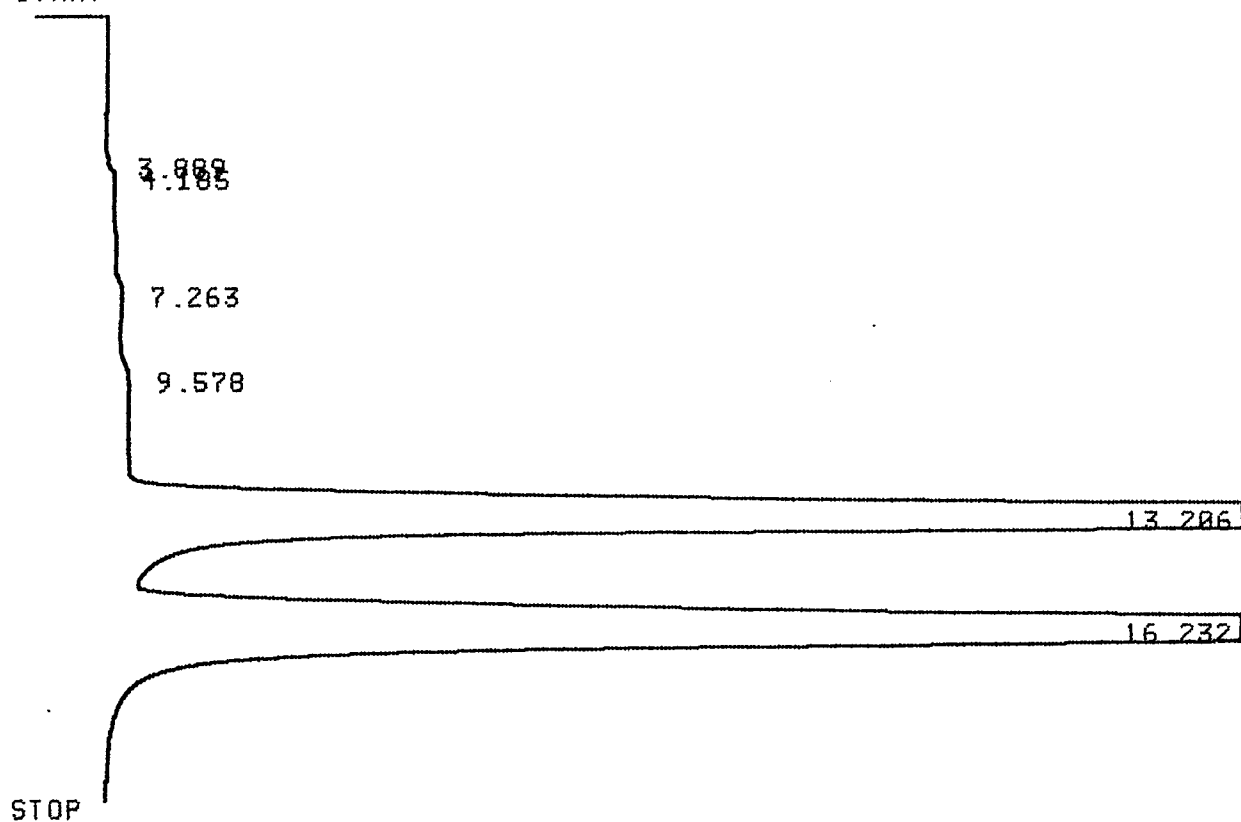
AREAX

RT	AREA	TYPE	WIDTH	AREAX
3.718	20128	BU	.111	.04433
3.862	32788	UU	.154	.07222
4.144	64306	UU	.248	.14164
8.327	114096	UU	1.002	.25130
13.196	45088064	UB	.642	99.30765
16.434	83034	BP	.713	.18288

TOTAL AREA=4.5402E+07
MUL FACTOR=1.0000E+00

HPLC 4**Racemic-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)**

* RUN # 1532 JUN 12, 1997 10:29:27
START



STOP

RUN# 1532 JUN 12, 1997 10:29:27

AREA%

RT	AREA	TYPE	WIDTH	AREA%
4.185	19923	PV	.253	.04316
7.263	39170	PV	.535	.08486
9.578	41623	PV	.466	.09018
13.206	23246000	PB	.667	50.36216
16.232	22810976	BB	.843	49.41966

TOTAL AREA=4.6158E+07

MUL FACTOR=1.0000E+00

Chiral HPLC Conditions for:

(*S*)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)

Chiralcel OD column (4.6 mm x 25 cm)

Mobile phase: 65:35 hexane/EtOH + 0.1% TFA

Flow rate: 1 mL/min

Detector: 335 nm

Injection size 10 µL

Retention times:

(*S*)-1, 13.2 min

(*R*)-1, 16.2 min.

Sample preparation

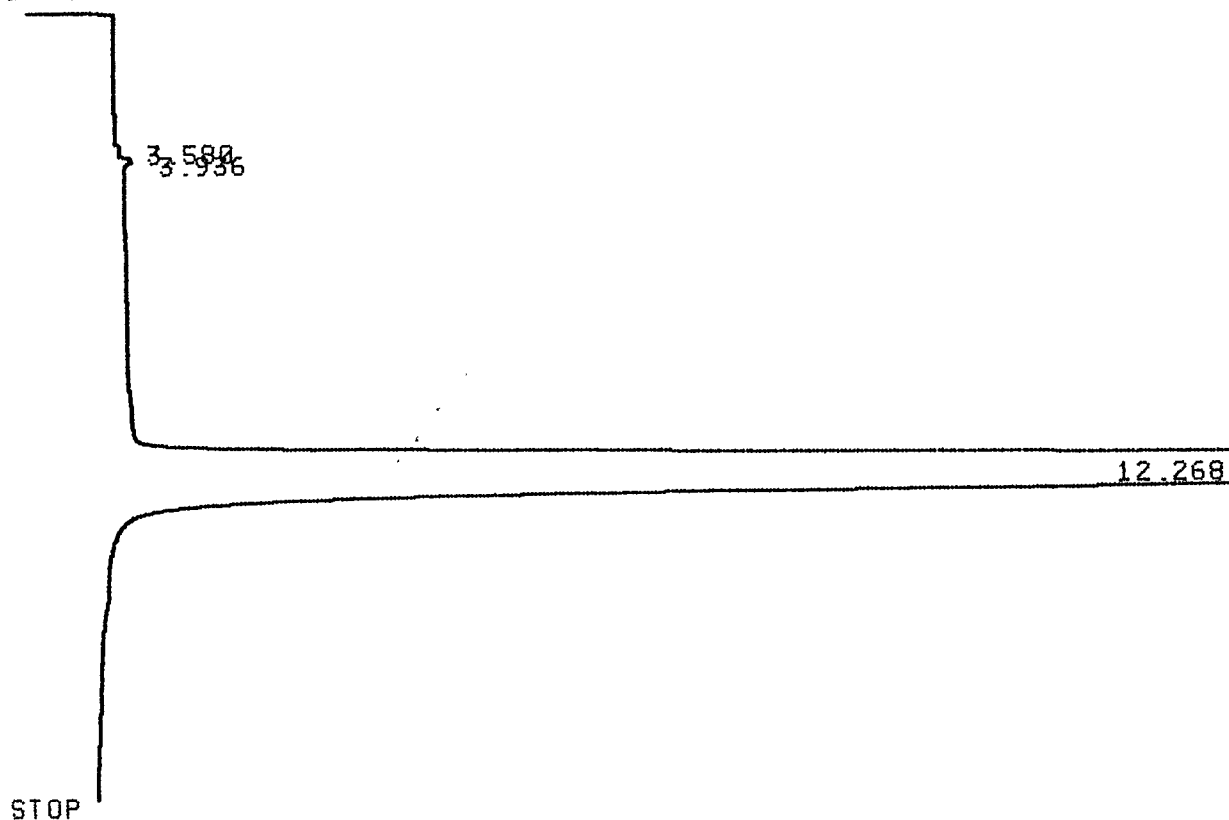
0.01g compound **1** dissolved in 5 mL ethanol and then diluted with 15 mL Mobile phase.

EE determination was made by dissolving 0.2502g compound **1** in 125 mL ethanol and diluting to a volume of 500 mL with the mobile phase. HPLC trace labelled 1 is a direct injection of this solution. The sample for HPLC trace 2 has 0.1% of *R*-1 added (1.0 mL of a solution of 0.0251g *R*-1 (ee >98%) dissolved in 100 mL ethanol). The sample for HPLC trace 3 has 0.2% *R*-1 added. HPLC trace 4 is racemic compound **1**.

HPLC 1

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)

* RUN # 1529 JUN 12, 1997 09:08:56
START



RUN# 1529 JUN 12, 1997 09:08:56

RT	AREA	TYPE	WIDTH	AREA%
3.580	10502	BU	.121	.02256
3.936	46066	UU	.200	.09898
12.268	46404960	UB	.646	99.87846

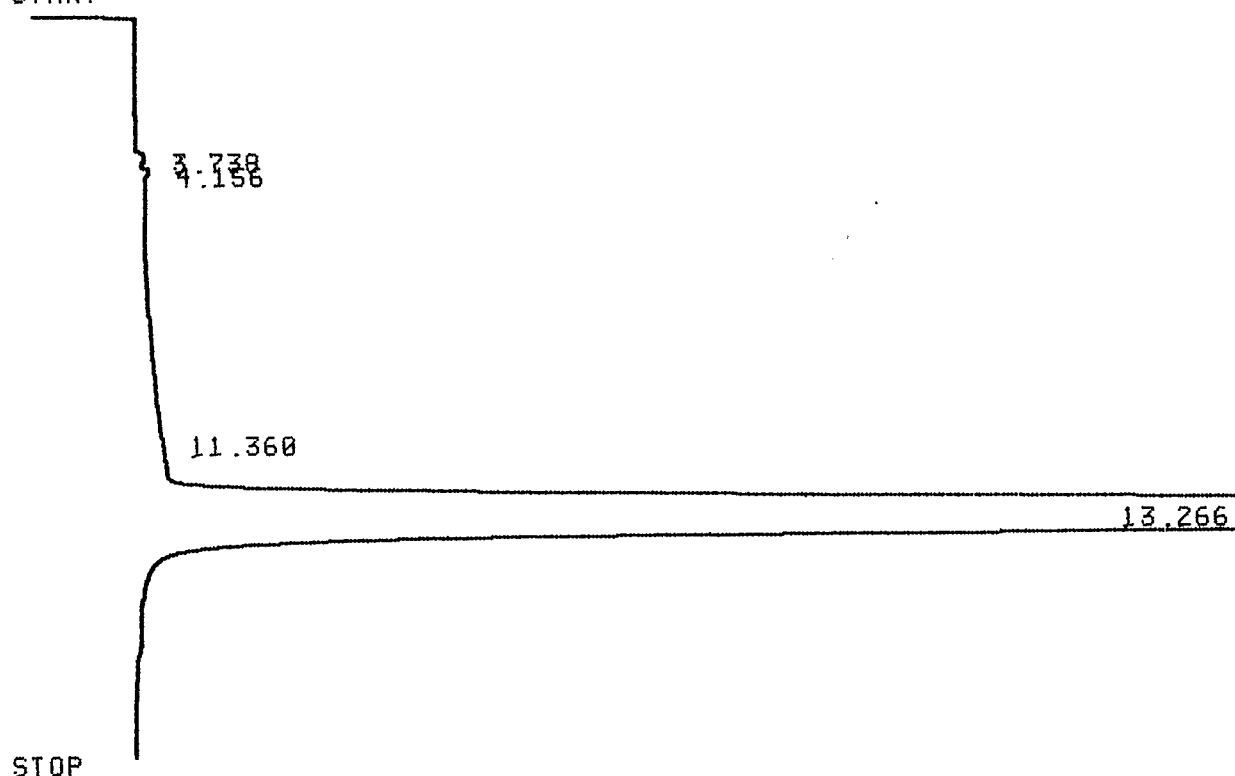
TOTAL AREA=4.6542E+07
MUL FACTOR=1.0000E+00

HPLC 2

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1) + 0.1% R Enantiomer

* RUN # 1530 JUN 12, 1997 09:47:11

START



RUN# 1530 JUN 12, 1997 09:47:11

AREA%

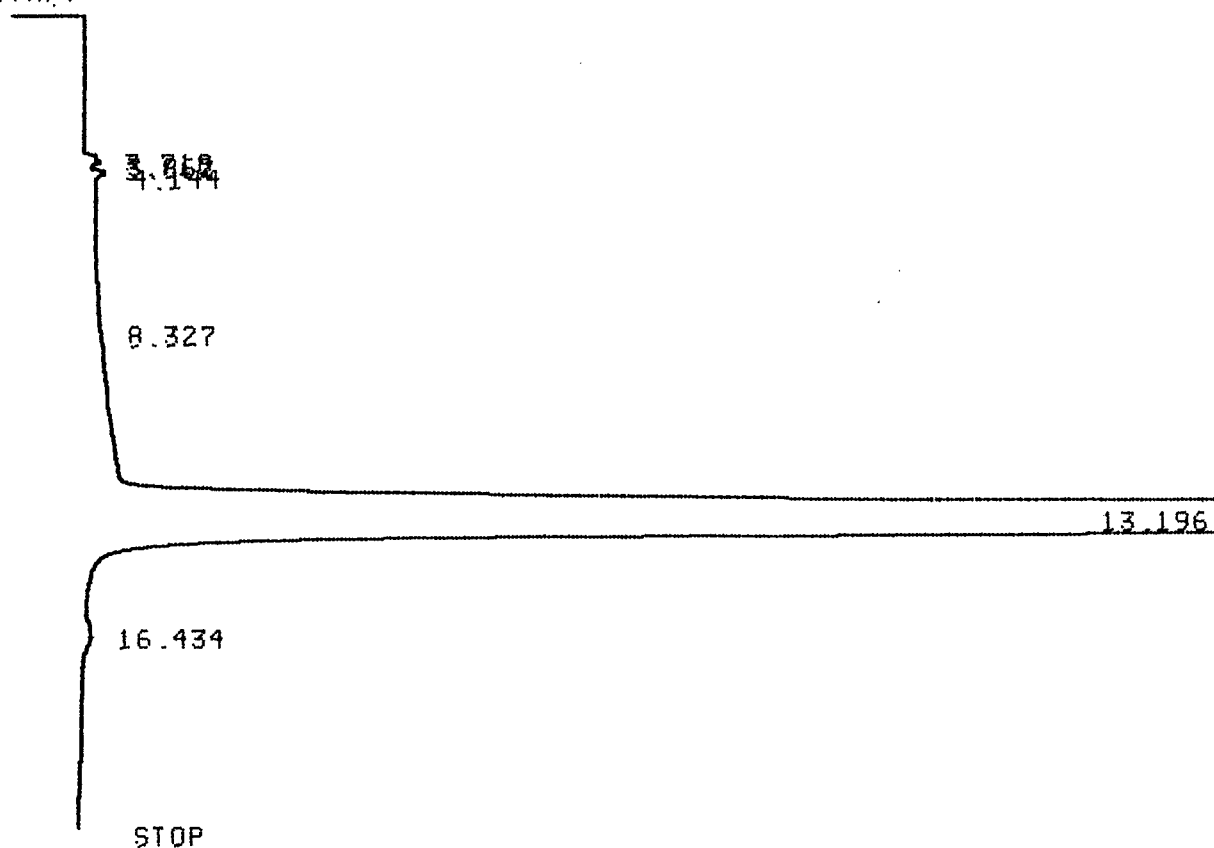
RT	AREA	TYPE	WIDTH	AREA%
3.738	14361	PV	.124	.03190
4.156	27102	VB	.193	.06020
11.360	131093	BV	.886	.29121
13.266	44843936	VB	.637	99.61670

TOTAL AREA=4.5016E+07
MUL FACTOR=1.0000E+00

HPLC 3

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1) + 0.2% R Enantiomer

* RUN # 1531 JUN 12, 1997 10:07:28
START



RUN# 1531 JUN 12, 1997 10:07:28

AREA%

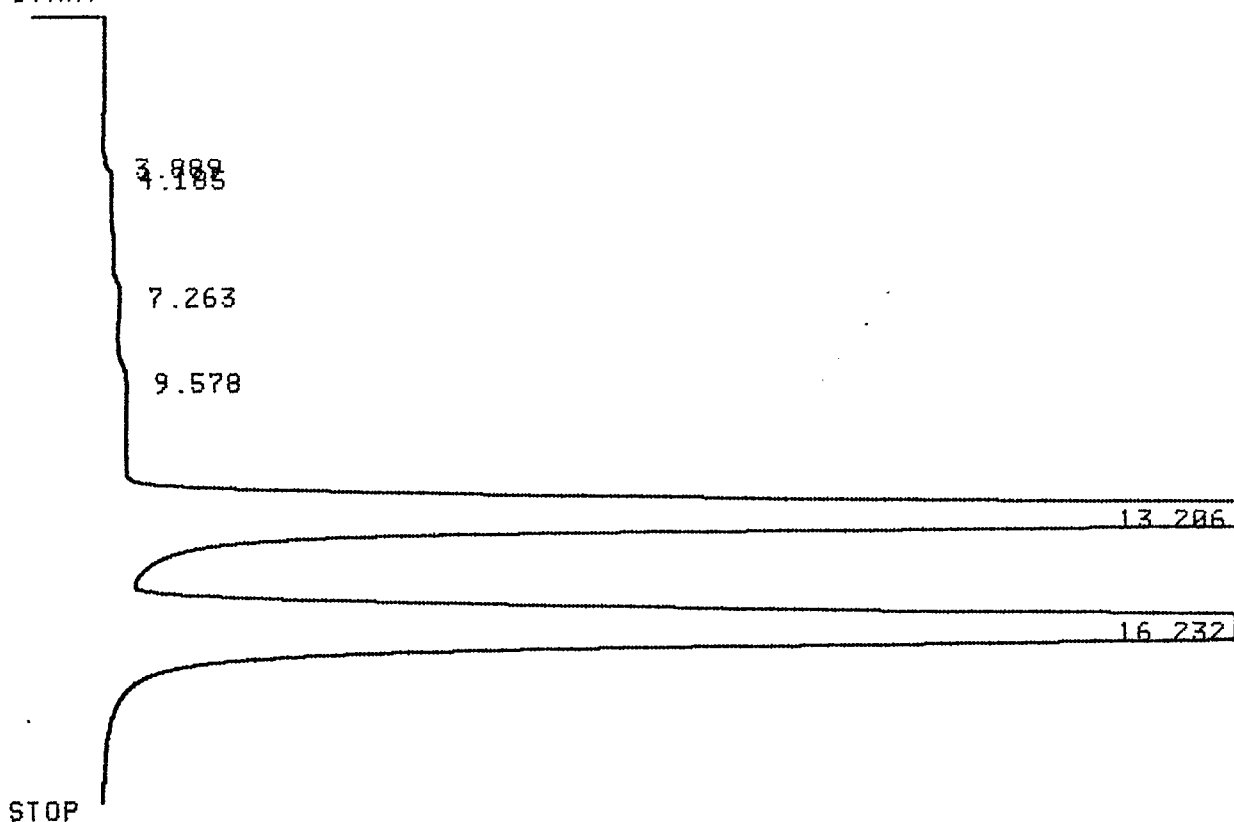
RT	AREA	TYPE	WIDTH	AREA%
3.718	20128	BU	.111	.04433
3.862	32788	UU	.154	.07222
4.144	64306	UU	.248	.14164
8.327	114096	UU	1.002	.25130
13.196	45088064	UB	.642	99.30765
16.434	83034	BP	.713	.18288

TOTAL AREA=4.5402E+07
MUL FACTOR=1.0000E+00

HPLC 4

Racemic-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)

* RUN # 1532 JUN 12, 1997 10:29:27
START



STOP

RUN# 1532 JUN 12, 1997 10:29:27

AREAX

RT	AREA	TYPE	WIDTH	AREAX
4.185	19923	PV	.253	.04316
7.263	39170	PV	.535	.08486
9.578	41623	PV	.466	.09018
13.206	23246000	PB	.667	50.36216
16.232	22810976	BB	.843	49.41966

TOTAL AREA=4.6158E+07

MUL FACTOR=1.0000E+00

Chiral HPLC Conditions for:

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)

Chiralcel OD column (4.6 mm x 25 cm)

Mobile phase: 65:35 hexane/EtOH + 0.1% TFA

Flow rate: 1 mL/min

Detector: 335 nm

Injection size 10 µL

Retention times:

(S)-1, 13.2 min

(R)-1, 16.2 min.

Sample preparation

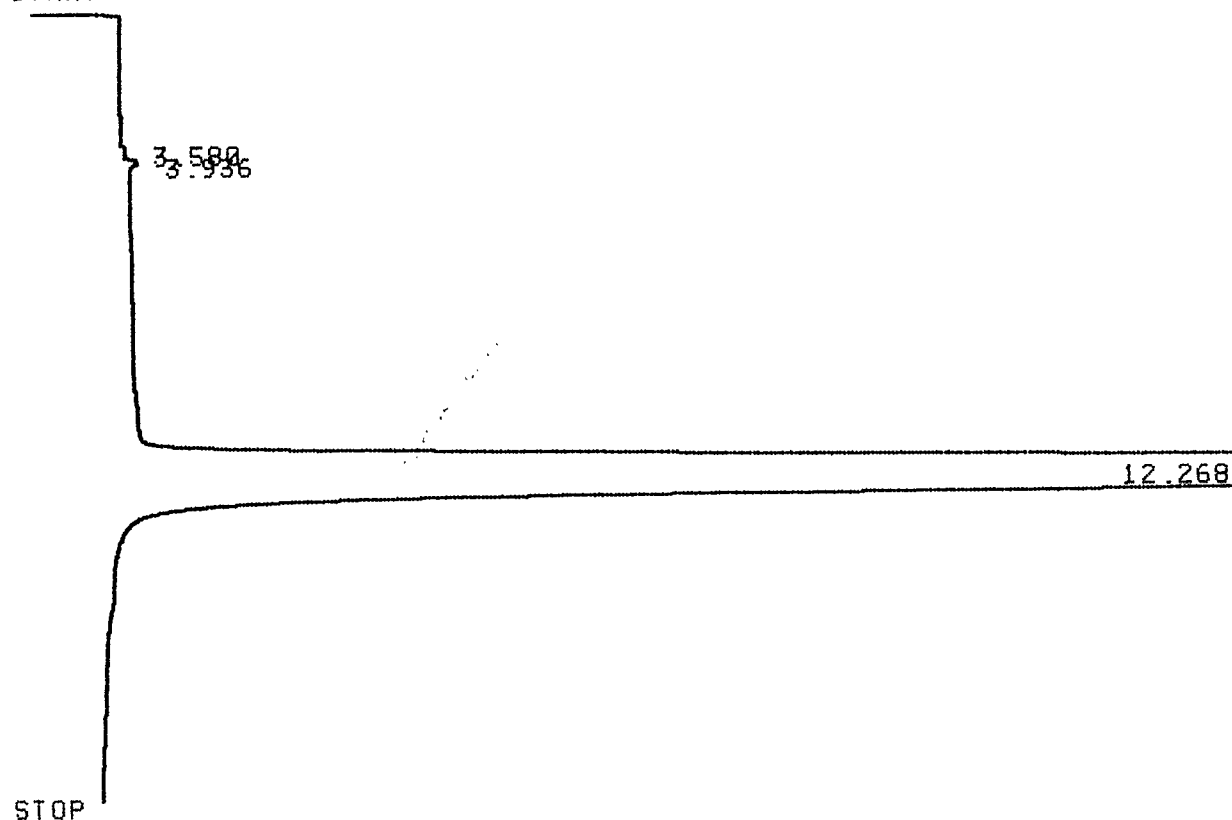
0.01g compound **1** dissolved in 5 mL ethanol and then diluted with 15 mL Mobile phase.

EE determination was made by dissolving 0.2502g compound **1** in 125 mL ethanol and diluting to a volume of 500 mL with the mobile phase. HPLC trace labelled 1 is a direct injection of this solution. The sample for HPLC trace 2 has 0.1% of **R-1** added (1.0 mL of a solution of 0.0251g **R-1** (ee >98%) dissolved in 100 mL ethanol). The sample for HPLC trace 3 has 0.2% **R-1** added. HPLC trace 4 is racemic compound **1**.

HPLC 1

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)

* RUN # 1529 JUN 12, 1997 09:08:56
START



RUN# 1529 JUN 12, 1997 09:08:56

AREA%

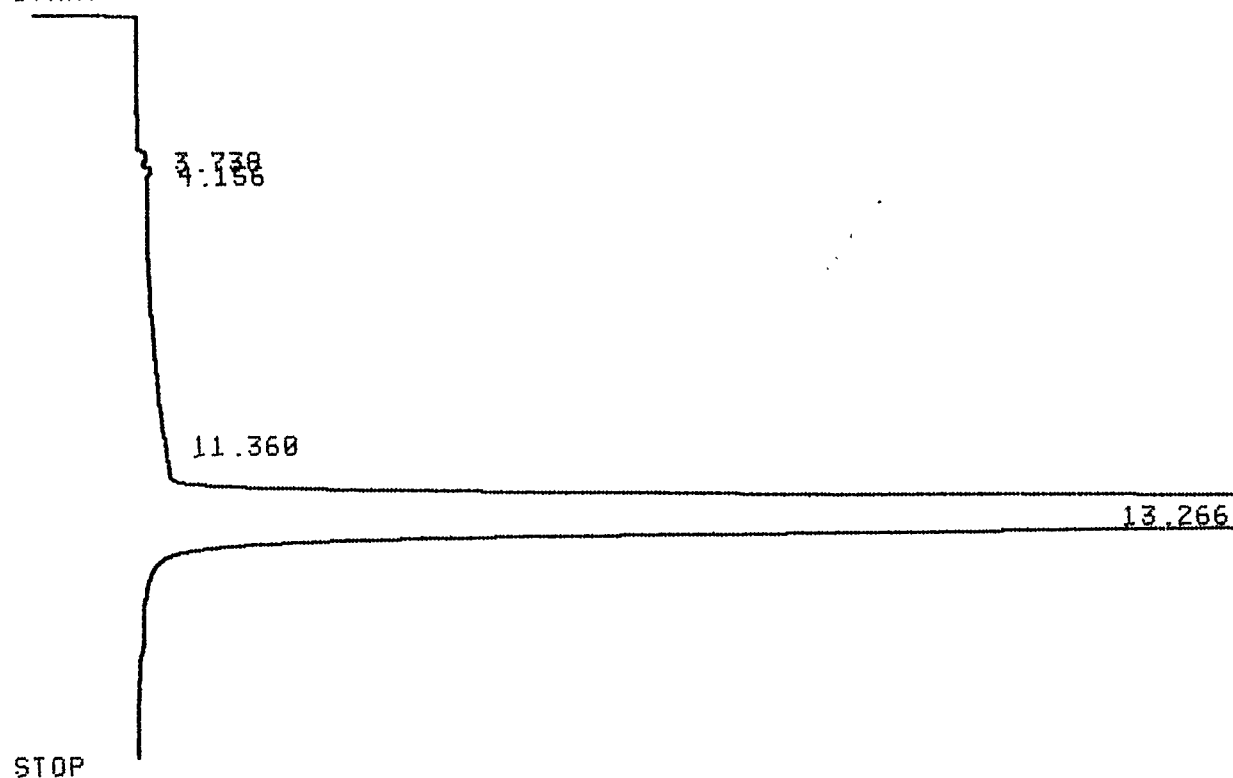
RT	AREA	TYPE	WIDTH	AREA%
3.580	10502	BU	.121	.02256
3.936	46066	UU	.200	.09898
12.268	46484960	UB	.646	99.87846

TOTAL AREA=4.6542E+07
MUL FACTOR=1.0000E+00

HPLC 2

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1) + 0.1% R Enantiomer

* RUN # 1530 JUN 12, 1997 09:47:11
START



RUN# 1530 JUN 12, 1997 09:47:11

AREA%

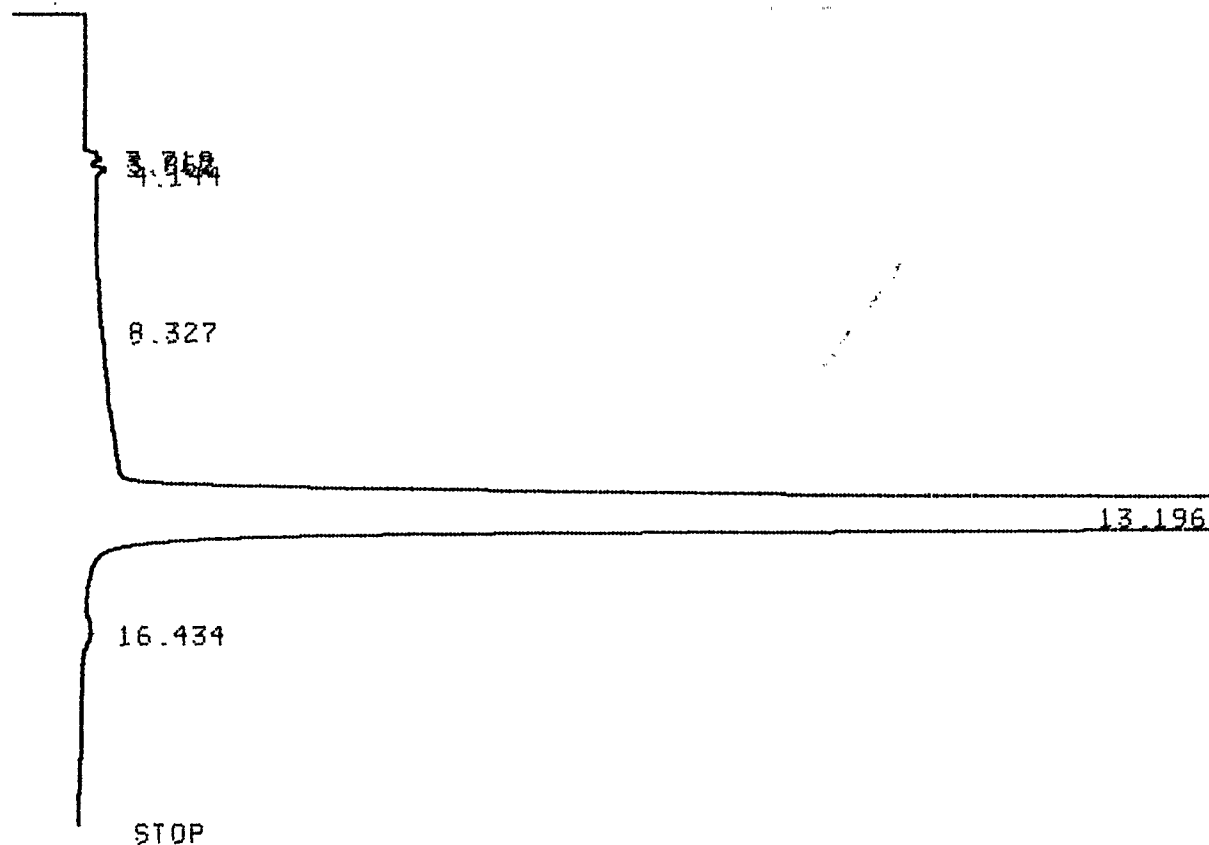
RT	AREA	TYPE	WIDTH	AREA%
3.738	14361	PV	.124	.03190
4.156	27102	VB	.193	.06020
11.360	131093	BV	.886	.29121
13.266	44843936	VB	.637	99.61670

TOTAL AREA=4.5016E+07
MUL FACTOR=1.0000E+00

HPLC 3

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-
[3,4-f]-indolizine (1) + 0.2% R Enantiomer

* RUN # 1531 JUN 12, 1997 10:07:28
START



RUN# 1531 JUN 12, 1997 10:07:28

AREA%

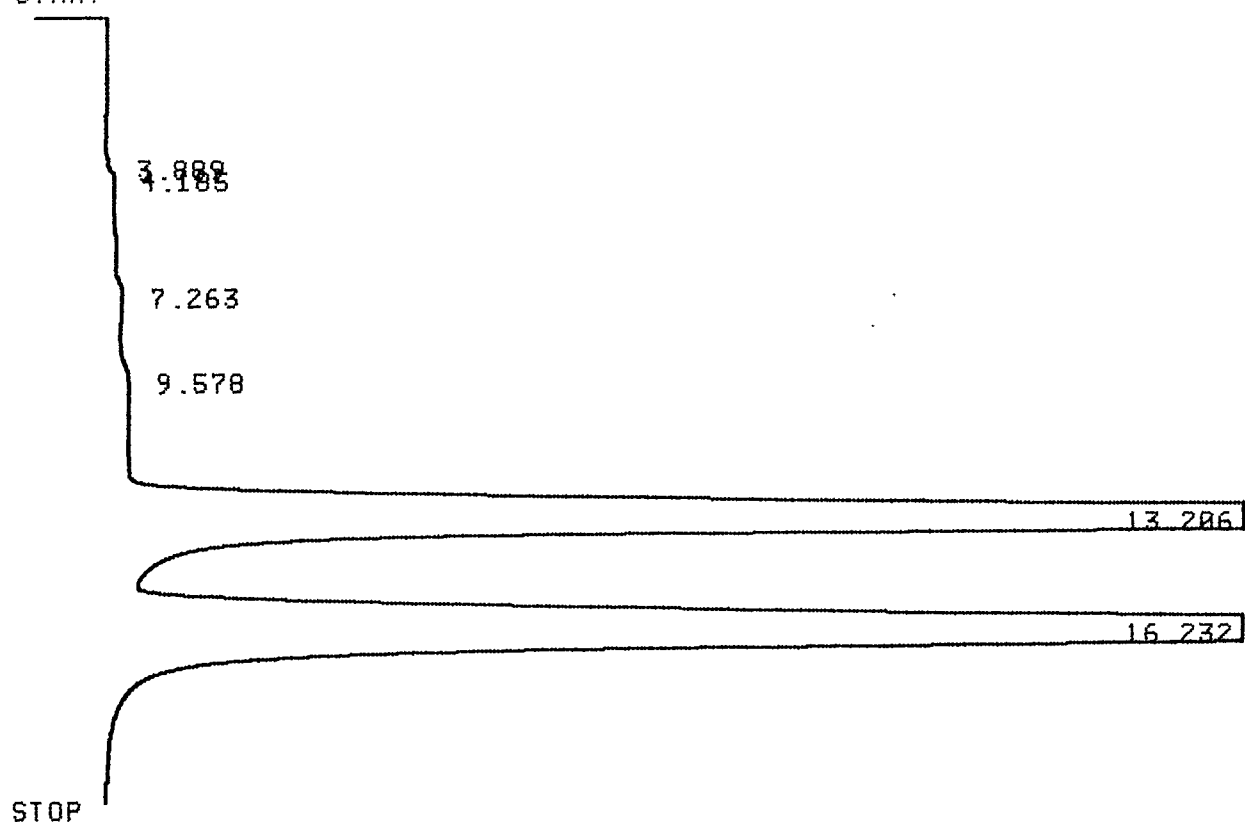
RT	AREA	TYPE	WIDTH	AREA%
3.718	20128	BU	.111	.04433
3.862	32788	UU	.154	.07222
4.144	64306	UU	.248	.14164
8.327	114096	UU	1.002	.25130
13.196	45088064	UB	.642	99.30765
16.434	83034	BP	.713	.18288

TOTAL AREA=4.5402E+07
MUL FACTOR=1.0000E+00

HPLC 4

Racemic-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)

* RUN # 1532 JUN 12, 1997 10:29:27
START



RUN# 1532 JUN 12, 1997 10:29:27

AREA%

RT	AREA	TYPE	WIDTH	AREA%
4.185	19923	PV	.253	.04316
7.263	39170	PV	.535	.08486
9.578	41623	PV	.466	.09018
13.206	23246000	PB	.667	50.36216
16.232	22810976	BB	.843	49.41966

TOTAL AREA=4.6158E+07

MUL FACTOR=1.0000E+00

Chiral HPLC Conditions for:

(*S*)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)

Chiralcel OD column (4.6 mm x 25 cm)

Mobile phase: 65:35 hexane/EtOH + 0.1% TFA

Flow rate: 1 mL/min

Detector: 335 nm

Injection size 10 µL

Retention times:

(*S*)-1, 13.2 min

(*R*)-1, 16.2 min.

Sample preparation

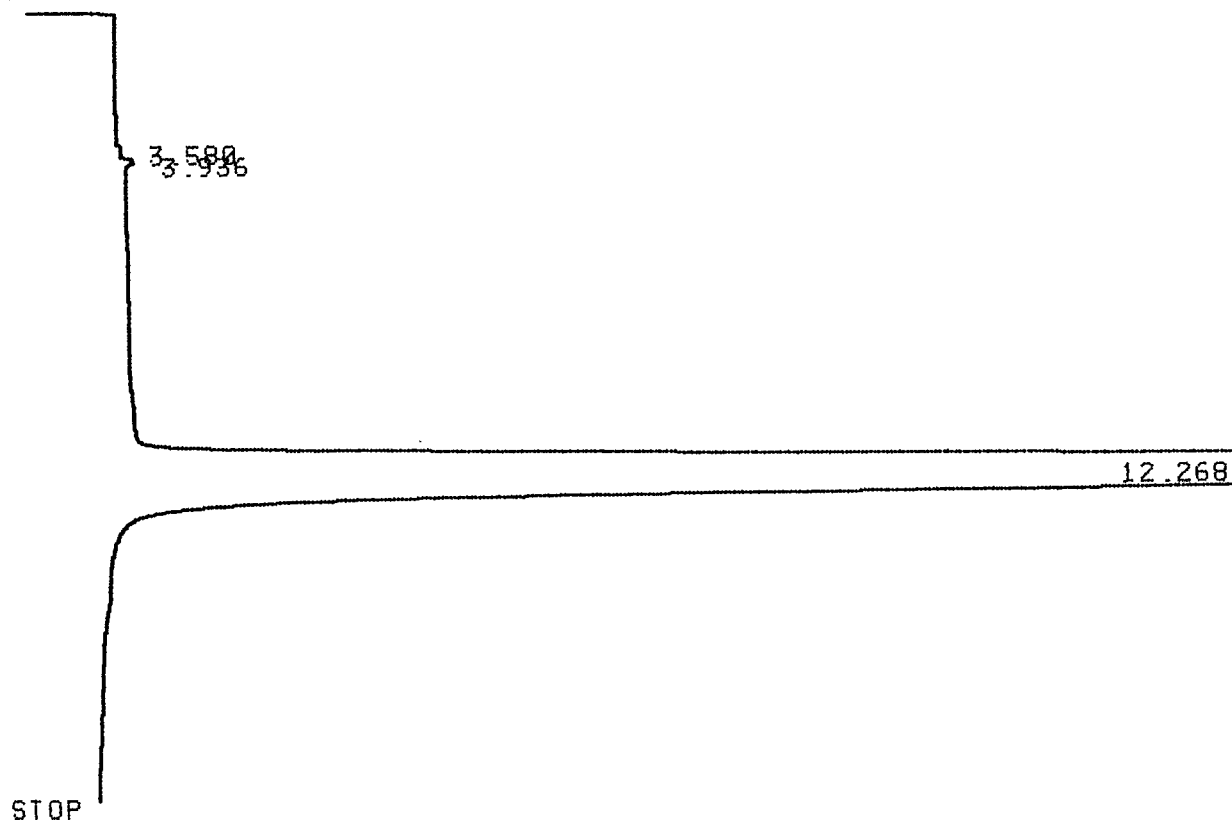
0.01g compound **1** dissolved in 5 mL ethanol and then diluted with 15 mL Mobile phase.

EE determination was made by dissolving 0.2502g compound **1** in 125 mL ethanol and diluting to a volume of 500 mL with the mobile phase. HPLC trace labelled 1 is a direct injection of this solution. The sample for HPLC trace 2 has 0.1% of *R*-**1** added (1.0 mL of a solution of 0.0251g *R*-**1** (ee >98%) dissolved in 100 mL ethanol). The sample for HPLC trace 3 has 0.2% *R*-**1** added. HPLC trace 4 is racemic compound **1**.

HPLC 1

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)

* RUN # 1529 JUN 12, 1997 09:08:56
START



RUN# 1529 JUN 12, 1997 09:08:56

AREA%

RT	AREA	TYPE	WIDTH	AREA%
3.580	10502	BU	.121	.02256
3.936	46066	UU	.200	.09898
12.268	46484960	UB	.646	99.87846

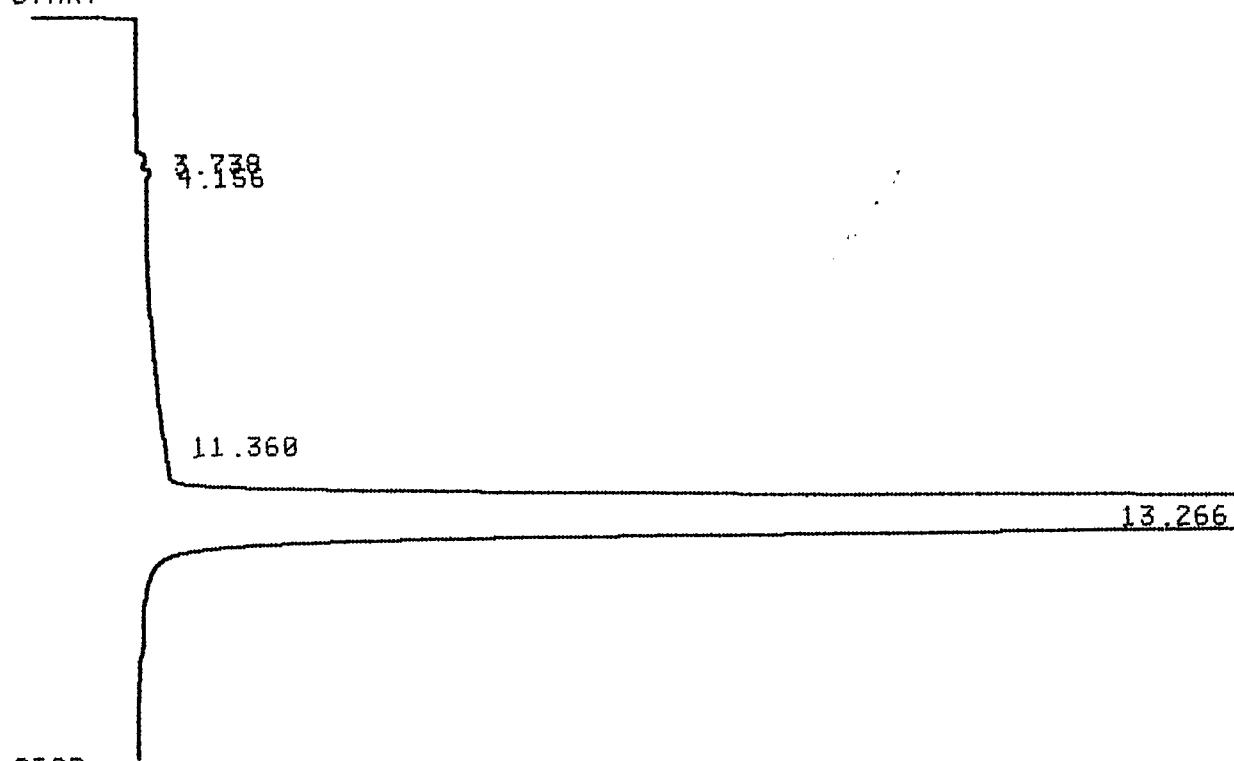
TOTAL AREA=4.6542E+07
MUL FACTOR=1.0000E+00

HPLC 2

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-
[3,4-f]-indolizine (1) + 0.1% R Enantiomer

* RUN # 1530 JUN 12, 1997 09:47:11

START



STOP

RUN# 1530 JUN 12, 1997 09:47:11

AREA%

RT	AREA	TYPE	WIDTH	AREA%
3.738	14361	PV	.124	.03190
4.156	27102	UB	.193	.06020
11.360	131093	BV	.886	.29121
13.266	44843936	UB	.637	99.61670

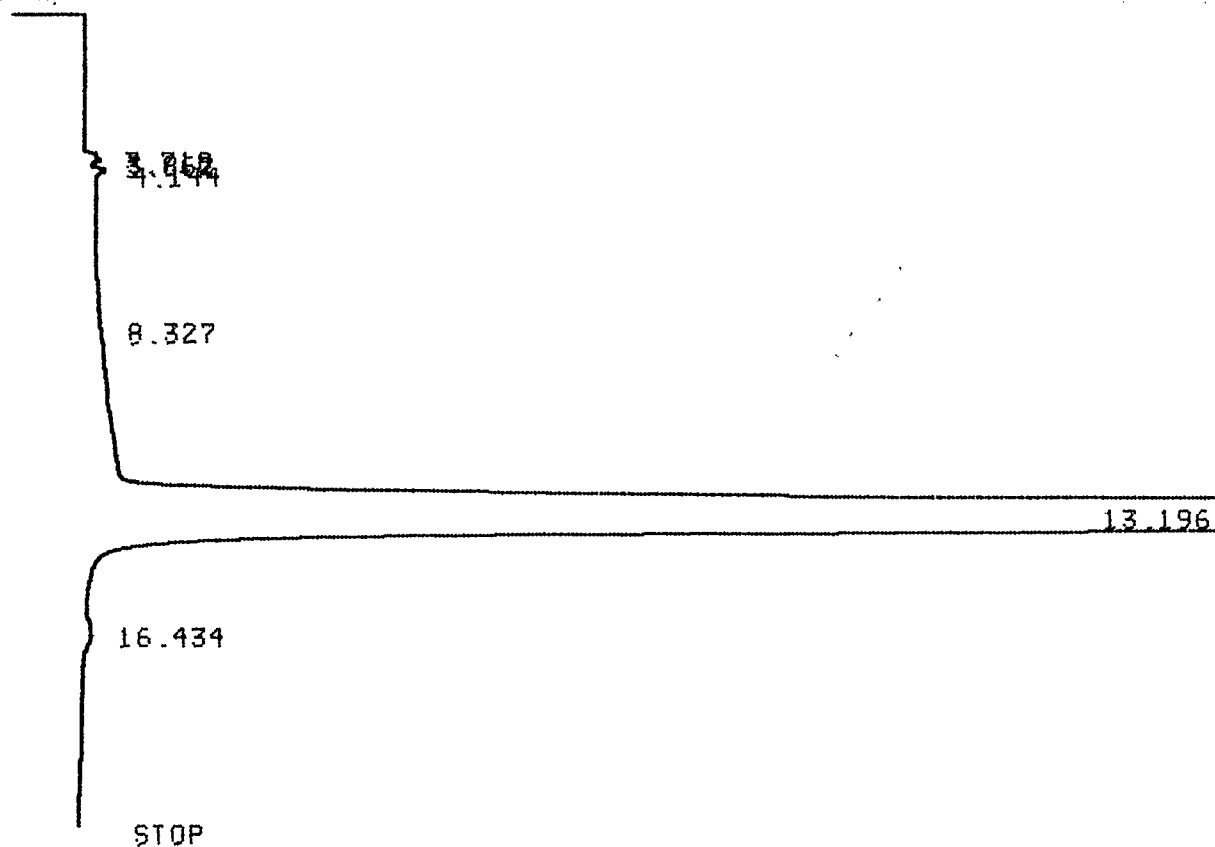
TOTAL AREA=4.5016E+07

MUL FACTOR=1.0000E+00

HPLC 3

(S)-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1) + 0.2% R Enantiomer

* RUN # 1531 JUN 12, 1997 10:07:28
START



RUN# 1531 JUN 12, 1997 10:07:28

AREA%

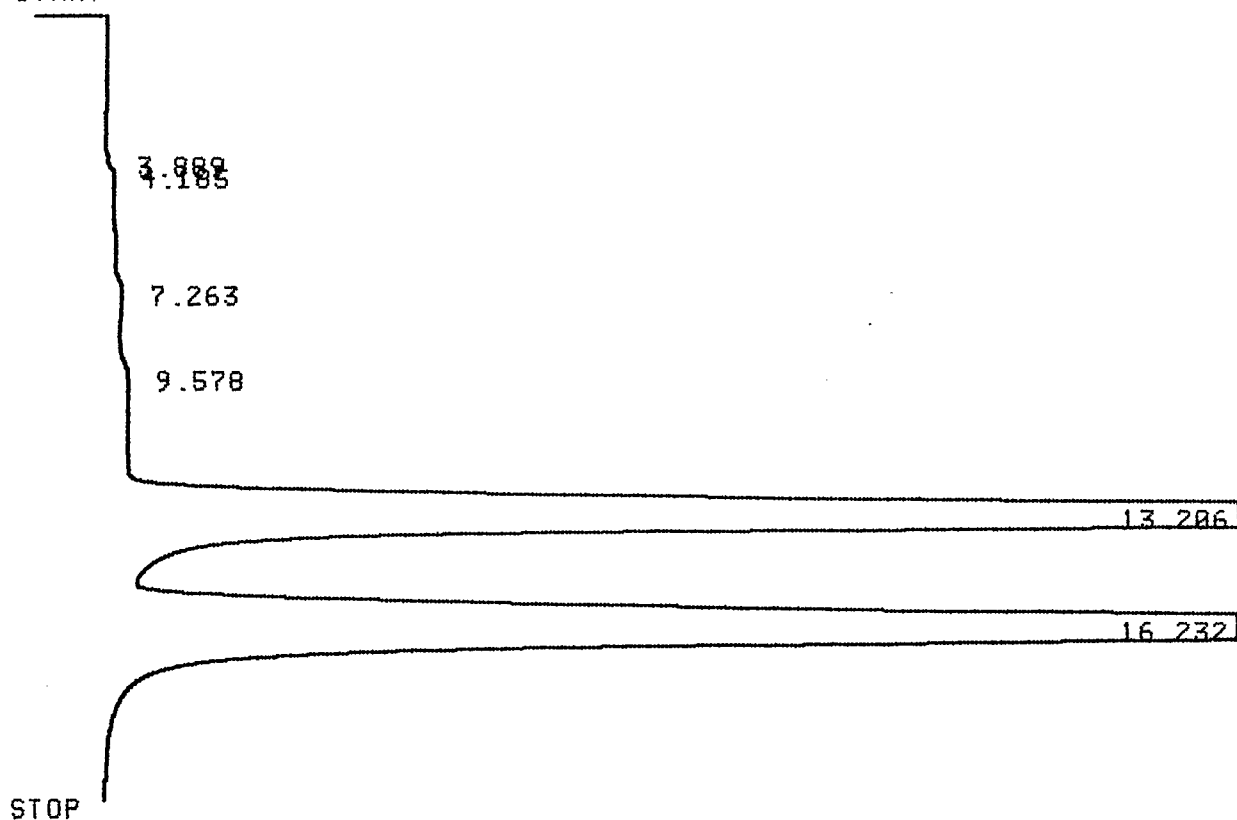
RT	AREA	TYPE	WIDTH	AREA%
3.718	20128	BU	.111	.04433
3.862	32788	UU	.154	.07222
4.144	64306	UU	.248	.14164
8.327	114096	UU	1.002	.25130
13.196	45088064	UB	.642	99.30765
16.434	83034	BP	.713	.18288

TOTAL AREA=4.5402E+07
MUL FACTOR=1.0000E+00

HPLC 4

Racemic-4-Ethyl-3,4,6,10(4H)-trione-4-ethyl-7,8-dihydro-4-hydroxy-1H-pyrano-[3,4-f]-indolizine (1)

* RUN # 1532 JUN 12, 1997 10:29:27
START



RUN# 1532 JUN 12, 1997 10:29:27

AREAX

RT	AREA	TYPE	WIDTH	AREAX
4.185	19923	PV	.253	.04316
7.263	39170	PV	.535	.08486
9.578	41623	PV	.466	.09018
13.206	23246000	PB	.667	50.36216
16.232	22810976	BB	.843	49.41966

TOTAL AREA=4.6158E+07
MUL FACTOR=1.0000E+00