

Reprint from

RECENT ADVANCES
IN DOPING ANALYSIS
(5)

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G. SIGMUND, S. HORNING, I. SEINSCH, W. SCHÄNZER, M. DONIKE:
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Confirmation of Clenbuterol Reextraction with Hydrochloric acid

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INTRODUCTION

Clenbuterol is applied in very small doses and so the concentration in urine is low. Therefore for confirmation an extract with low biological background is needed. The following sample preparation is very useful to reduce disturbing coelution and to improve the detection limit.

SAMPLE PREPARATION

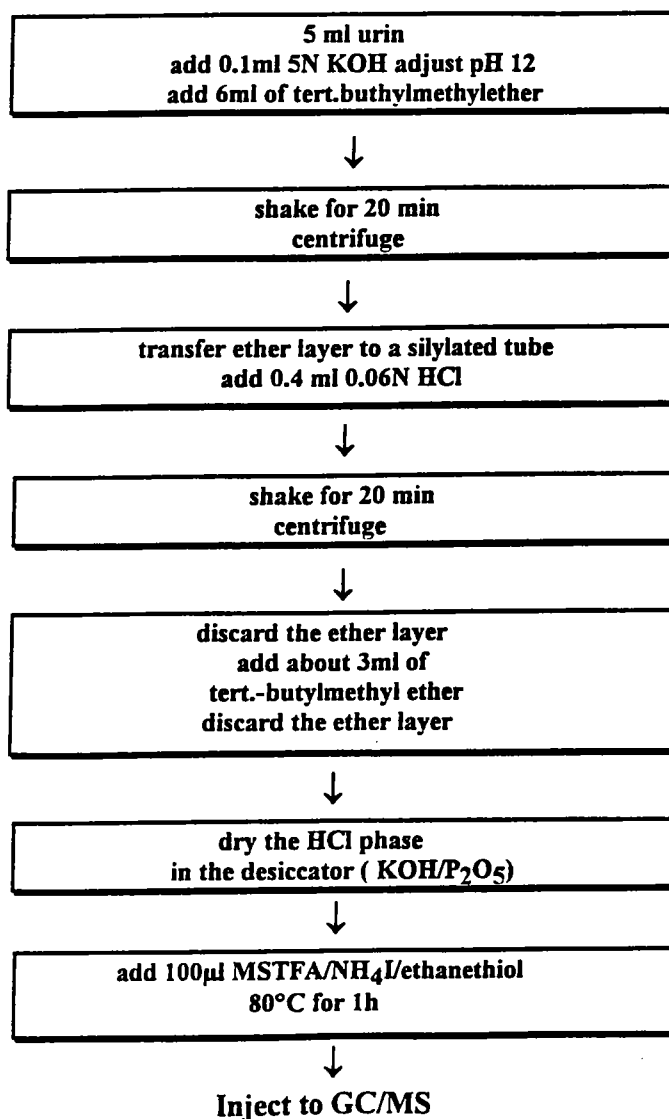


Figure 1
Sample preparation

RESULTS

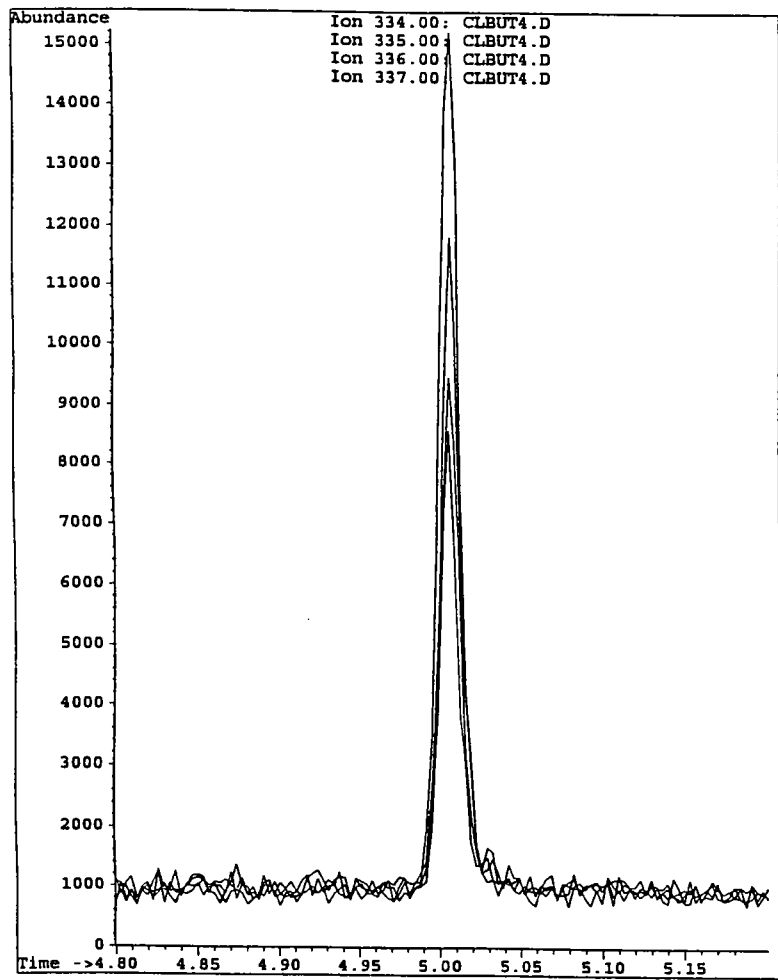


Figure 2
2 ng/ml clenbuterol
reextracted urine
GC/MS SIM

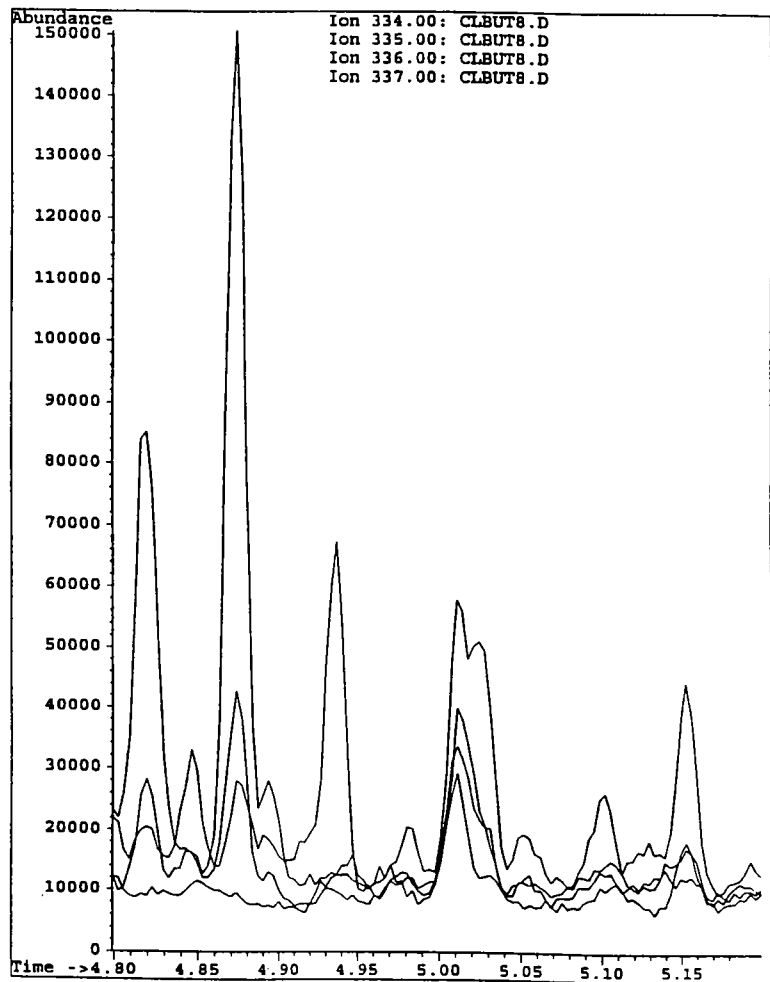


Figure 3
2 ng/ml clenbuterol
anab. steroids combined fraction
GC/MS SIM

Figure 4
 0.2 ng/ml clenbuterol
 reextracted urine
 GC/HRMS SIM

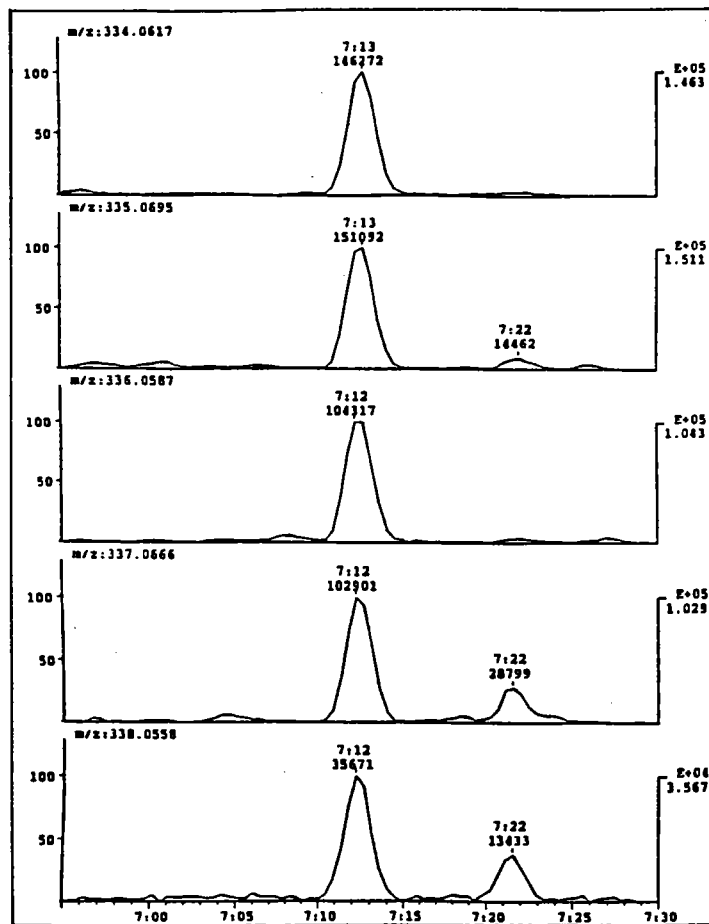
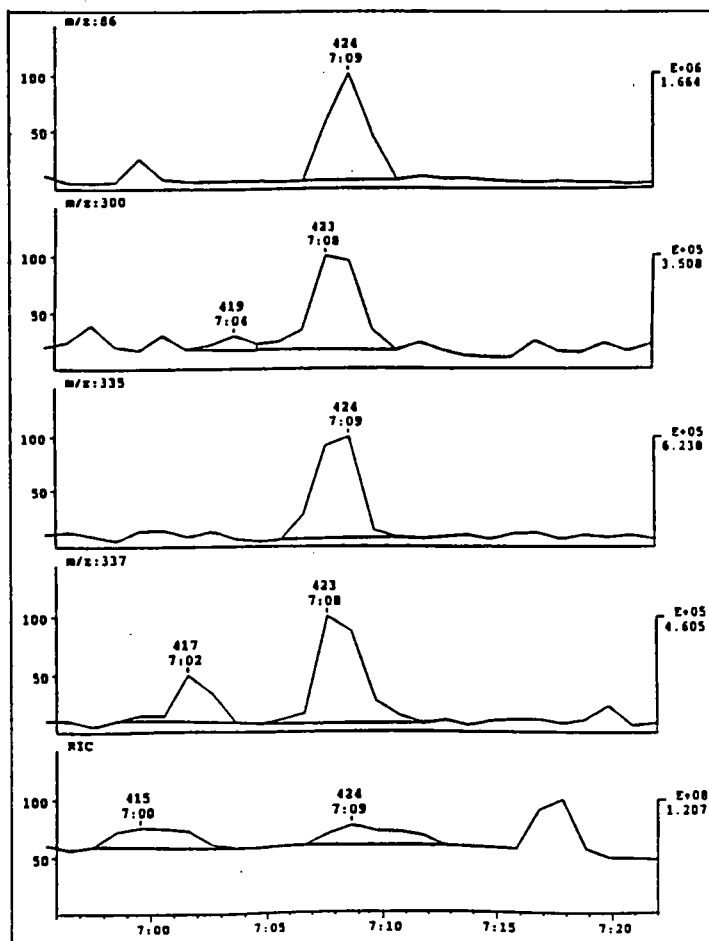


Figure 5
 2 ng/ml clenbuterol
 reextracted urine
 GC/HRMS SCAN



ISTD: Cloranolol [20 ng/ml]

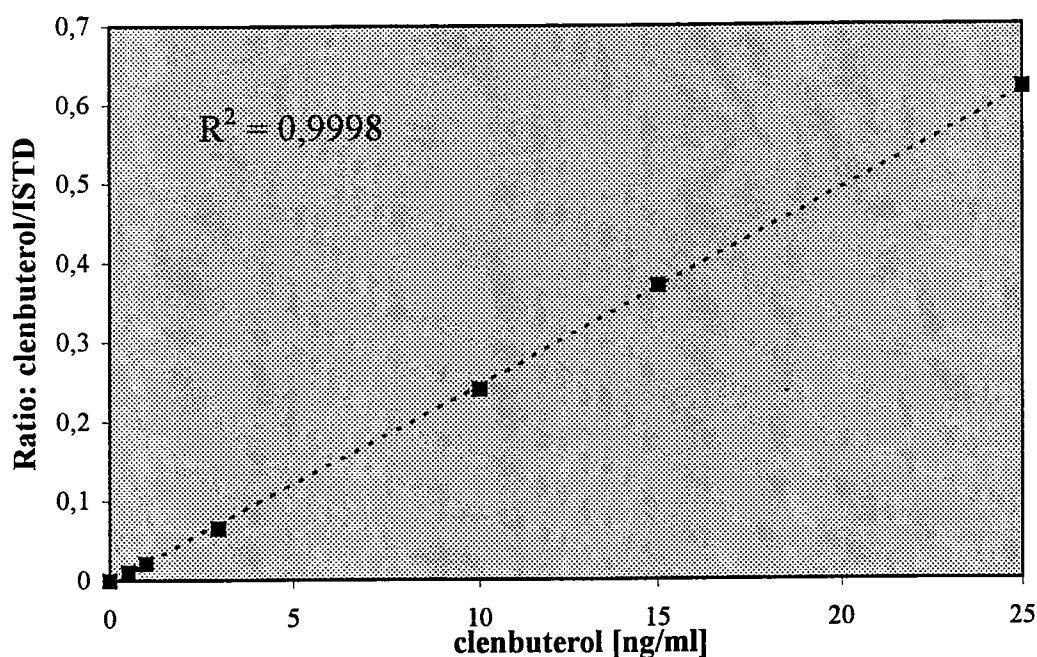


Figure 6
Clenbuterol calibration curve
in urine

Recovery: 86%
Reproducibility: 3.6% (n=6)

GC/MS Parameters

GC/HRMS-System: Hewlett Packard 5890/ Finnigan MAT 95
Carrier gas: Helium (1ml/min flow)
Split ratio: 1:20
Analytical column: HP-Ultra-1 fused silica capillary column,
crosslinked methylsilicone (OV 1), 16m,
0.2 mm i.D., 0.11 µm film thickness
Temperature program: 130°C, 10°C/min till 210°C, 40°C/min till 310°C
Mass resolution: 3500

GC/MSD-System: Hewlett Packard 5890/5971A
Carrier gas: Helium (1ml/min flow)
Split ratio: 1:10
Analytical column: HP-5 MS fused silica capillary column, 20m,
0.25 mm i.D., 0.25 µm film thickness
Temperature program: 130°C, 22°C/min till 320°C, 1 min constant

CONCLUSION

The biological background is greatly reduced with this sample preparation. Confirmation of clenbuterol concentration as low as 2 ng/ml in urine is possible in SCAN-mode. Lower concentrations (ca. 20 pg/ml) can be confirmed with HRMS/SIM-analysis.

LITERATURE

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