13 LIS 2007

Warszawa, dnia .....

GŁÓWNY INSPEKTOR SANITARNY

Andrzej Wojtyła

GIS-BŻ-Ż-4230-1155/JS//07

**Państwowy Wojewódzki Inspektor Sanitarny**

**-wszyscy-**

Szanowni Państwo,

W związku z informacją Narodowego Instytutu Leków w sprawie ujawnienia sfałszowanych suplementów diety uprzejmie proszę o podjęcie w tym zakresie działań zgodnych z ustawowymi kompetencjami.

W załączeniu przekazuję do wiadomości i wykorzystania służbowego 18 raportów o produktach sfałszowanych (suplementach diety) otrzymanych z Europejskiego Dyrektoriatu Jakości Leków w Strasbourgu (EDQM – OMCL Network).

Informacje o wyniku przeprowadzonych działań wyjaśniających proszę przesłać niezwłocznie do Głównego Inspektora Sanitarnego.

Z poważaniem

Główny Inspektor Sanitarny
z up.
Przemysław Bilński
Zastępca Głównego Inspektora Sanitarnego

ZAŁĄCZNIKI WYSŁANO POCZTĄ ELEKTRONICZNĄ
- 18 RAPORTÓW

# Information Exchange of Counterfeit/Illegal Medicines in the OMCL Network

## Part 1. Organisation details

| | |
|---|---|
| Competent Authority/OMCL | Medical Products Agency, Uppsala, Sweden, MPA SE |
| Contact Person | Ann-Kristin Arvidsson |
| Telephone N° (Include National Code) | +46 18 17 47 92 |
| E-Mail Address | ann-kristin.arvidsson@mpa.se |

## Part 2. Product Details

| | |
|---|---|
| Laboratory Reference Number | Dno 297:2006/34260 |
| Product Origin i.e. Internet, Legal/Illegal Market etc | Internet (Universalnutrition.se) |
| Product Name | Thermo Stack |
| Labelled Active Ingredient | Guarana extract (22 % caffeine) and Bitter Orange (4% synephrine) |
| Licensed/Unlicensed | Unlicensed |
| Product Type | Dietary supplement |
| Dosage form (tablet, capsule, etc.) | Capsules |
| Strength per unit dose | Coffeine 44 mg (calculated from the declared value of Guarana)<br>Synephrine 4 mg (calc. from the declare value of Bitter orange.) |
| Pack size | Plastic jar, 90 capsules |
| MAHolder/Manufacturer (Where Appropriate) | Universal Nutrition<br>New brunwick, USA |
| Distributor (If Different) | N/A |
| Batch Number | 151864 |
| Expiry Date | 11/30/09 |
| Rapid alert reference number | N/A |

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|---|---|
| **Summary of Problem or Deficiency**<br>**i.e.**<br>**Provide details of analysis results.**<br>**Include Illegal actives identified**<br>**If reporting counterfeit products provide information on packaging livery and any visual identifiers and any results obtained on authentic comparators** | The product was tested for ephedrine, pseudoephedrine, synephrine, coffeine, fenfluramine, nitrosofenfluramine, sibutramine, orlistate, levothyroxine, liothyronin, dexamphetamine, usnic acid, using NMR, HPLC, GC-MS and LC-MS. Also the content of 2,4-dinitrophenol, methylphenidate, hordenine, bergenin, salicin and green tea was checked using NMR.<br>**NMR and HPLC- analyses showed that the product contained 22 mg coffeine and 5 mg synephrine per capsule. The other substances were not found.** |
| **Detail of Analytical Method Used**<br>**i.e. HPLC, Column type, Mobile phase and Detection system. Info regarding availability of reference materials** | **1. ID and Assay by HPLC – UV**<br>**(ephedrine, pseudoephedrine, synephrine, coffeine, fenfluramine)**<br><br>Column: Zorbax SB Phenyl 5 µm; 250 x 4,6 mm<br>Eluent A: 0,2 M Ammonium acetate buffer pH 4,8<br>Eluent B: Acetonitrile/ 0,2 M ammonium acetate buffer pH 4,8 (90/10% v/v)<br>Flow: 1,0 ml/min<br>Column temperature: 25°C<br>Injection volume: 20 µl<br><br>Gradient: 5,5% B for 15 min; 5,5% B to 48% B in 15 min; 48% B for 5 min; 48% B to 80% B in 2 min; 80% B for 5 min; 80% B to 5,5 % B in 1 min; 5,5% B for 12 min<br><br>Detection: PDA 200-350 nm,<br>1 spectrum/sec; 1,2 nm resolution.<br><br>**2. ID and Assay by $^{1}H$ and $^{13}C$ NMR spectroscopy. Bruker Avance 600 MHz.**<br><br>**3. ID by GC-MS**<br>**(Presence of fenfluramine, nitrosofenfluramine and amphetamine)**<br><br>Instrument: Agilent Technologies 6890N Network GC Systems<br>Detector: Agilent 5973 N Network Mass Selective<br>Column: HP-5MSi 30m*0,25mm, film thickness 0,25µm<br>Injector temp: 250°C |

| | |
|---|---|
| | Injection volume: 1µl<br>Split 10:1<br>Carrier gas: Helium<br>Flow: 1mL/min<br>Owen temp: 60°C (1min) 10°C/min 300°C (35min)<br>Aux: 280°C<br>MS Quad: 150°C<br>MS Source: 230°C<br>Scan: m/z 40-400<br>SIM: m/z 159, 186<br><br>**4. ID by LC-MS**<br>**( Presence of levothyroxine and liothyronin)**<br><br>HPLC-instrument: Waters Acquity UPLC<br>MS-instrument: Waters/Micromass Quattro II with Z-spray (electrospray)<br>Column: Zorbax Eclipse XDB-C18, 3,5 µm, 2,1 x 150 mm<br>Eluent A: 0,01 M ammonium acetate (with respect to ammonium) pH 4,5<br>Eluent B: 1 % av 1M ammonium acetate (aq) pH 4,5 + 5% methanol + 94% acetonitrile<br>Flow: 0,2 mL/min<br>Gradient: 5,5 % B to 48% B in 20 min; 48% B to 90% B in 2 min; 5 min at 90% B<br>Injection volume: 5 µl<br>Column temperature: 40°C<br>PDA: 210 – 350 nm; 2 spectra/second; 1,2 nm resolution<br>MS: Electrospray, negative mode (ES-)<br>MS-detection: SIR channel for m/z 649,7 with dwell time 0,20 and cone volt 30<br>SIR channel for m/z 775,5 with dwell time 0,20 and cone volt 30 |
| **Further actions taken by the OMCL** | **The product is included in a project where several weight reduction products bought via Internet were screened. Actions taken by the Swedish authority, MPA, are**<br>**-information to the media and**<br>**-report to the police for products where ephedrine or pseudoephedrine have been found.** |
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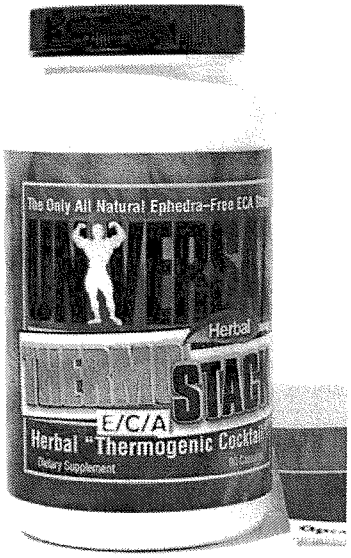


Dnr 297:2006/34260

# Information Exchange of Counterfeit/Illegal Medicines in the OMCL Network

## Part 1. Organisation details

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| Competent Authority/OMCL | Medical Products Agency, Uppsala, Sweden, MPA SE |
| Contact Person | Ann-Kristin Arvidsson |
| Telephone N° (Include National Code) | +46 18 17 47 92 |
| E-Mail Address | ann-kristin.arvidsson@mpa.se |

## Part 2. Product Details

| | |
|---|---|
| Laboratory Reference Number | Dno 297:2006/24868 |
| Product Origin i.e. Internet, Legal/Illegal Market etc | Internet (Bantning.nu) |
| Product Name | Termoxical |
| Labelled Active Ingredient | Usnic acid |
| Licensed/Unlicensed | Unlicensed |
| Product Type | Dietary supplement |
| Dosage form (tablet, capsule, etc.) | Tablets |
| Strength per unit dose | Usnic acid 10 mg |
| Pack size | Plastic jar, 120 tablets |
| MAHolder/Manufacturer (Where Appropriate) | (Pure Natural Herbs) Web address www.smartlab.dk |
| Distributor (If Different) | N/ |
| Batch Number | N/A |
| Expiry Date | N/A |
| Rapid alert reference number | N/A |

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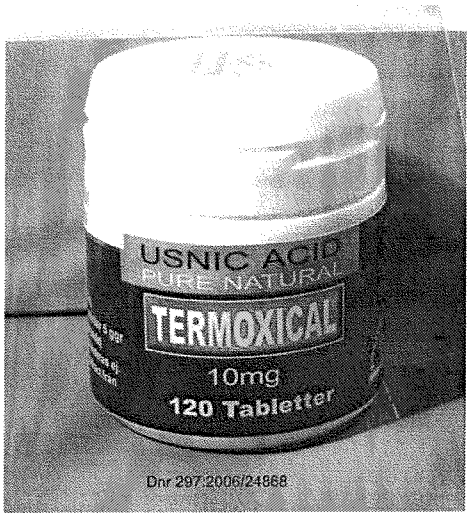
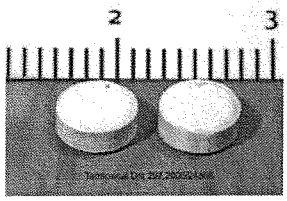
|---|---|
| **Summary of Problem or Deficiency**<br>**i.e.**<br>**Provide details of analysis results.**<br>**Include Illegal actives identified**<br>**If reporting counterfeit products provide information on packaging livery and any visual identifiers and any results obtained on authentic comparators** | The product was tested for ephedrine, pseudoephedrine, synephrine, coffeine, fenfluramine, nitrosofenfluramine, sibutramine, orlistate, levothyroxine, liothyronin, dexamphetamine, usnic acid, using NMR, HPLC, GC-MS and LC-MS. Also the content of 2,4-dinitrophenol, methylphenidate, hordenine, bergenin, salicin and green tea was checked using NMR.<br>**NMR analyses showed that the product contained 9,7 mg usnic acid. The tablets also contained about 0,02 mg synephrine, detected by NMR. The other substances were not found.** |
| **Detail of Analytical Method Used**<br>**i.e. HPLC, Column type, Mobile phase and Detection system. Info regarding availability of reference materials** | **1. ID and Assay by HPLC – UV**<br>**(ephedrine, pseudoephedrine, synephrine, coffeine, fenfluramine)**<br><br>Column: Zorbax SB Phenyl 5 µm; 250 x 4,6 mm<br>Eluent A: 0,2 M Ammonium acetate buffer pH 4,8<br>Eluent B: Acetonitrile/ 0,2 M ammonium acetate buffer pH 4,8 (90/10% v/v)<br>Flow: 1,0 ml/min<br>Column temperature: 25°C<br>Injection volume: 20 µl<br><br>Gradient: 5,5% B for 15 min; 5,5% B to 48% B in 15 min; 48% B for 5 min; 48% B to 80% B in 2 min; 80% B for 5 min; 80% B to 5,5 % B in 1 min; 5,5% B for 12 min<br><br>Detection: PDA 200-350 nm,<br>1 spectrum/sec; 1,2 nm resolution.<br><br>**2. ID and Assay by $^{1}H$ and $^{13}C$ NMR spectroscopy. Bruker Avance 600 MHz.**<br><br>**3. ID by GC-MS**<br>**(Presence of fenfluramine, nitrosofenfluramine and amphetamine)**<br><br>Instrument: Agilent Technologies 6890N Network GC Systems<br>Detector: Agilent 5973 N Network Mass Selective<br>Column: HP-5MSi 30m*0,25mm, film thickness 0,25µm |

| | |
|---|---|
| | Injector temp: 250°C<br>Injection volume: 1µl<br>Split 10:1<br>Carrier gas: Helium<br>Flow: 1mL/min<br>Owen temp: 60°C (1min) 10°C/min 300°C (35min)<br>Aux: 280°C<br>MS Quad: 150°C<br>MS Source: 230°C<br>Scan: m/z 40-400<br>SIM: m/z 159, 186<br><br>**4. ID by LC-MS**<br>**( Presence of levothyroxine and liothyronin)**<br><br>HPLC-instrument: Waters Acquity UPLC<br>MS-instrument: Waters/Micromass Quattro II with Z-spray (electrospray)<br>Column: Zorbax Eclipse XDB-C18, 3,5 µm, 2,1 x 150 mm<br>Eluent A: 0,01 M ammonium acetate (with respect to ammonium) pH 4,5<br>Eluent B: 1 % av 1M ammonium acetate (aq) pH 4,5 + 5% methanol + 94% acetonitrile<br>Flow: 0,2 mL/min<br>Gradient: 5,5 % B to 48% B in 20 min; 48% B to 90% B in 2 min; 5 min at 90% B<br>Injection volume: 5 µl<br>Column temperature: 40°C<br>PDA: 210 – 350 nm; 2 spectra/second; 1,2 nm resolution<br>MS: Electrospray, negative mode (ES-)<br>MS-detection: SIR channel for m/z 649,7 with dwell time 0,20 and cone volt 30<br>SIR channel for m/z 775,5 with dwell time 0,20 and cone volt 30 |
| **Further actions taken by the OMCL** | **The product is included in a project where several weight reduction products bought via Internet were screened. Actions taken by the Swedish authority, MPA, are**<br>**-information to the media and**<br>**-report to the police for products where ephedrine or pseudoephedrine have been found.** |
| | |


USNIC ACID
PURE NATURAL
TERMOXICAL
10mg
120 Tabletter
Dnr 297:2006/24868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# Information Exchange of Counterfeit/Illegal Medicines in the OMCL Network

## Part 1. Organisation details

| | |
|---|---|
| Competent Authority/OMCL | Medical Products Agency, Uppsala, Sweden, MPA SE |
| Contact Person | Ann-Kristin Arvidsson |
| Telephone N° (Include National Code) | +46 18 17 47 92 |
| E-Mail Address | ann-kristin.arvidsson@mpa.se |

## Part 2. Product Details

| | |
|---|---|
| Laboratory Reference Number | Dno 297:2006/34162 |
| Product Origin i.e. Internet, Legal/Illegal Market etc | Internet (Tdcs.se) |
| Product Name | Synerlin |
| Labelled Active Ingredient | Synephrine, coffeine |
| Licensed/Unlicensed | Unlicensed |
| Product Type | Dietary supplement |
| Dosage form (tablet, capsule, etc.) | Tablets |
| Strength per unit dose | Synephrine 75mg, Coffeine 125 mg |
| Pack size | Plastic jar, 120 tablets |
| MAHolder/Manufacturer (Where Appropriate) | Self omninutrition, 13:e protein import, Sverige |
| Distributor (If Different) | N/A |
| Batch Number | 4020 |
| Expiry Date | 03/2009 |
| Rapid alert reference number | N/A |

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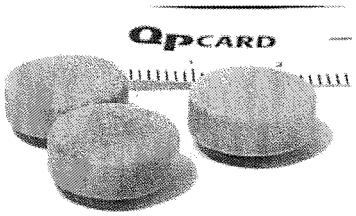
|---|---|
| **Summary of Problem or Deficiency**<br>**i.e.**<br>**Provide details of analysis results.**<br>**Include Illegal actives identified**<br>**If reporting counterfeit products provide information on packaging livery and any visual identifiers and any results obtained on authentic comparators** | The product was tested for ephedrine, pseudoephedrine, synephrine, coffeine, fenfluramine, nitrosofenfluramine, sibutramine, orlistate, levothyroxine, liothyronin, dexamphetamine, usnic acid, using NMR, HPLC, GC-MS and LC-MS. Also the content of 2,4-dinitrophenol, methylphenidate, hordenine, bergenin, salicin and green tea was checked using NMR.<br>**NMR analysis showed that the product contained 2 mg ephedrine and 2 mg pseudoephedrine per tablet. NMR and HPLC analyses showed that the product contained 248 mg coffeine per tablet. NMR spectra also indicated content of green tea, which was declared. The other substances were not found.** |
| **Detail of Analytical Method Used**<br>**i.e. HPLC, Column type, Mobile phase and Detection system. Info regarding availability of reference materials** | **1. ID and Assay by HPLC – UV**<br>**(ephedrine, pseudoephedrine, synephrine, coffeine, fenfluramine)**<br><br>Column: Zorbax SB Phenyl 5 µm; 250 x 4,6 mm<br>Eluent A: 0,2 M Ammonium acetate buffer pH 4,8<br>Eluent B: Acetonitrile/ 0,2 M ammonium acetate buffer pH 4,8 (90/10% v/v)<br>Flow: 1,0 ml/min<br>Column temperature: 25°C<br>Injection volume: 20 µl<br><br>Gradient: 5,5% B for 15 min; 5,5% B to 48% B in 15 min; 48% B for 5 min; 48% B to 80% B in 2 min; 80% B for 5 min; 80% B to 5,5 % B in 1 min; 5,5% B for 12 min<br><br>Detection: PDA 200-350 nm,<br>1 spectrum/sec; 1,2 nm resolution.<br><br>**2. ID and Assay by $^{1}H$ and $^{13}C$ NMR spectroscopy. Bruker Avance 600 MHz.**<br><br>**3. ID by GC-MS**<br>**(Presence of fenfluramine, nitrosofenfluramine and amphetamine)**<br><br>Instrument: Agilent Technologies 6890N Network GC Systems |

| | |
|---|---|
| | Detector: Agilent 5973 N Network Mass Selective<br>Column: HP-5MSi 30m*0,25mm, film thickness 0,25µm<br>Injector temp: 250°C<br>Injection volume: 1µl<br>Split 10:1<br>Carrier gas: Helium<br>Flow: 1mL/min<br>Owen temp: 60°C (1min) 10°C/min 300°C (35min)<br>Aux: 280°C<br>MS Quad: 150°C<br>MS Source: 230°C<br>Scan: m/z 40-400<br>SIM: m/z 159, 186<br><br>**4. ID by LC-MS**<br>**( Presence of levothyroxine and liothyronin)**<br><br>HPLC-instrument: Waters Acquity UPLC<br>MS-instrument: Waters/Micromass Quattro II with Z-spray (electrospray)<br>Column: Zorbax Eclipse XDB-C18, 3,5 µm, 2,1 x 150 mm<br>Eluent A: 0,01 M ammonium acetate (with respect to ammonium) pH 4,5<br>Eluent B: 1 % av 1M ammonium acetate (aq) pH 4,5 + 5% methanol + 94% acetonitrile<br>Flow: 0,2 mL/min<br>Gradient: 5,5 % B to 48% B in 20 min; 48% B to 90% B in 2 min; 5 min at 90% B<br>Injection volume: 5 µl<br>Column temperature: 40°C<br>PDA: 210 – 350 nm; 2 spectra/second; 1,2 nm resolution<br>MS: Electrospray, negative mode (ES-)<br>MS-detection: SIR channel for m/z 649,7 with dwell time 0,20 and cone volt 30<br>SIR channel for m/z 775,5 with dwell time 0,20 and cone volt 30 |
| **Further actions taken by the OMCL** | **The product is included in a project where several weight reduction products bought via Internet were screened. Actions taken by the Swedish authority, MPA, are**<br>**-information to the media and**<br>**-report to the police for products where ephedrine or pseudoephedrine have been found.** |
| | |

Dnr 297:2006/34162




Dnr 297:2006/34162

# Information Exchange of Counterfeit/Illegal Medicines in the OMCL Network

## Part 1. Organisation details

| | |
|---|---|
| Competent Authority/OMCL | Medical Products Agency, Uppsala, Sweden, MPA SE |
| Contact Person | Ann-Kristin Arvidsson |
| Telephone N° (Include National Code) | +46 18 17 47 92 |
| E-Mail Address | ann-kristin.arvidsson@mpa.se |

## Part 2. Product Details

| | |
|---|---|
| Laboratory Reference Number | Dno 297:2006/24726 |
| Product Origin i.e.<br>Internet, Legal/Illegal Market etc | Internet (Topformula.se) |
| Product Name | Synephrine, Top Formula |
| Labelled Active Ingredient | Synephrine 200 mg |
| Licensed/Unlicensed | Unlicensed |
| Product Type | Dietary supplement |
| Dosage form (tablet, capsule, etc.) | Tablets |
| Strength per unit dose | Synephrine 200 mg |
| Pack size | Plastic jar, 100 tablets |
| MAHolder/Manufacturer<br>(Where Appropriate) | N/A |
| Distributor (If Different) | N/A |
| Batch Number | N/A |
| Expiry Date | N/A |
| Rapid alert reference number | N/A |
| Summary of Problem or Deficiency<br>i.e.<br>Provide details of analysis results.<br>Include Illegal actives identified<br>If reporting counterfeit products provide information on packaging livery and any visual identifiers and any results obtained on authentic comparators | The product was tested for ephedrine, pseudoephedrine, synephrine, coffeine, fenfluramine, nitrosofenfluramine, sibutramine, orlistate, levothyroxine, liothyronin, dexamphetamine, usnic acid, using NMR, HPLC, GC-MS and LC-MS. Also the content of 2,4-dinitrophenol, methylphenidate, hordenine, bergenin, salicin and green tea was checked using NMR.<br>**NMR and HPLC analyses showed that the product contained 192 mg synephrine.**<br>**NMR spectra indicated the presence of Hoodia, which was not declared. The other substances were not found.** |
| Detail of Analytical Method Used | 1. ID and Assay by HPLC – UV |

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|---|---|
| **i.e. HPLC, Column type, Mobile phase and Detection system. Info regarding availability of reference materials** | **(ephedrine, pseudoephedrine, synephrine, coffeine, fenfluramine)**<br><br>Column: Zorbax SB Phenyl 5 µm; 250 x 4,6 mm<br>Eluent A: 0,2 M Ammonium acetate buffer pH 4,8<br>Eluent B: Acetonitrile/ 0,2 M ammonium acetate buffer pH 4,8 (90/10% v/v)<br>Flow: 1,0 ml/min<br>Column temperature: 25°C<br>Injection volume: 20 µl<br><br>Gradient: 5,5% B for 15 min; 5,5% B to 48% B in 15 min; 48% B for 5 min; 48% B to 80% B in 2 min; 80% B for 5 min; 80% B to 5,5 % B in 1 min; 5,5% B for 12 min<br><br>Detection: PDA 200-350 nm,<br>1 spectrum/sec; 1,2 nm resolution.<br><br>**2. ID and Assay by $^{1}H$ and $^{13}C$ NMR spectroscopy. Bruker Avance 600 MHz.**<br><br>**3. ID by GC-MS**<br>**(Presence of fenfluramine, nitrosofenfluramine and amphetamine)**<br><br>Instrument: Agilent Technologies 6890N Network GC Systems<br>Detector: Agilent 5973 N Network Mass Selective<br>Column: HP-5MSi 30m*0,25mm, film thickness 0,25µm<br>Injector temp: 250°C<br>Injection volume: 1µl<br>Split 10:1<br>Carrier gas: Helium<br>Flow: 1mL/min<br>Owen temp: 60°C (1min) 10°C/min 300°C (35min)<br>Aux: 280°C<br>MS Quad: 150°C<br>MS Source: 230°C<br>Scan: m/z 40-400<br>SIM: m/z 159, 186<br><br>**4. ID by LC-MS**<br>**( Presence of levothyroxine and liothyronin)** |

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|---|---|
| | HPLC-instrument: Waters Acquity UPLC<br>MS-instrument: Waters/Micromass Quattro II with Z-spray (electrospray)<br>Column: Zorbax Eclipse XDB-C18, 3,5 µm, 2,1 x 150 mm<br>Eluent A: 0,01 M ammonium acetate (with respect to ammonium) pH 4,5<br>Eluent B: 1 % av 1M ammonium acetate (aq) pH 4,5 + 5% methanol + 94% acetonitrile<br>Flow: 0,2 mL/min<br>Gradient: 5,5 % B to 48% B in 20 min; 48% B to 90% B in 2 min; 5 min at 90% B<br>Injection volume: 5 µl<br>Column temperature: 40°C<br>PDA: 210 – 350 nm; 2 spectra/second; 1,2 nm resolution<br>MS: Electrospray, negative mode (ES-)<br>MS-detection: SIR channel for m/z 649,7 with dwell time 0,20 and cone volt 30<br>SIR channel for m/z 775,5 with dwell time 0,20 and cone volt 30 |
| **Further actions taken by the OMCL** | **The product is included in a project where several weight reduction products bought via Internet were screened. Actions taken by the Swedish authority, MPA, are**<br>**-information to the media and**<br>**-report to the police for products where ephedrine or pseudoephedrine have been found.** |
| | |

TOP FORMULA
SYNEPHRINE
200 MG
Synephrine Dnr 297:2006/24726

Synephrine Dnr 297:2006/24726