

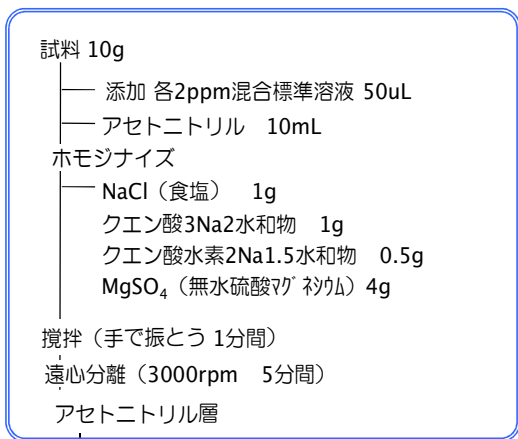
牛乳 (ギ酸メタノール使用)

MANUAL-LC



STQ-KIT
For STQ Method

前処理フロー



分取 1 mL

Smart-SPE C18-30 mg+PSA-30 mg : 精製

洗液 0.4%ギ酸メタノール 1mL (pH2.5)

流出液

水 0.5mL

Smart-SPE C18-50mg : 精製

溶出 メタノール-水 (8/2) 1mL

溶出液

定容 (4mL, 水で調製: 4倍希釈)

LC/MS/MS (注入量 5 uL : 試料 1.25mg相当)

目的/実験方法

手作業による添加回収試験(LC法)

- 添加濃度 (試料中) : 10ppb
- 最終バイアル中濃度 : 2.5ppb
- 農薬 : 関東MIX-53, 54, 55, 58, 45
(2ppmMIX/MeOHを作成)
: 一部のGC対象高極性農薬
(2ppmMIX/アセトニトリルを作成)
- 検量線 :
・ 絶対、直線検量線
・ 1点 : 2.5ppb
農薬混合標準溶液 (アセトニトリル : 80%メタノール/
水 : 0.4%ギ酸メタノール : 水 = 1:1:1:1)
- 測定 : sMRM測定 (1チャンネル/成分)



遠心分離後の状態

Sample



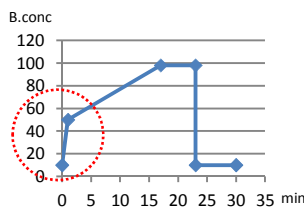
Information

水分 : 87.4%
脂質 : 3.8%
脂肪酸 : 3.32%

牛乳を検体とする。

LC/MS/MS分析条件

| | |
|-----------|---|
| 装置 | MS: API 3200Q system (AB SCIEX) |
| | LC: Prominence (SIMADZU) |
| 分析カラム | L-column2 ODS 2.1×150mm 3.0μm (化学物質評価研究機構) |
| 移動相 | A: 0.5mM酢酸アンモニウム水溶液 B: 0.5mM酢酸アンモニウム含有メタノール |
| グラジエント条件 | B conc. (%) |
| Positive | 10%→50% (0-1min) →98% (1-17min) →98% (17-23min) →10% (23-30min) |
| Negative | 10%→50% (0-2min) →98% (2-10min) →98% (10-13min) →10% (13-20min) |
| 分析時間 | 30min (Positive), 20min (Negative) |
| 流速 | 0.2mL/min |
| 注入量 | 5μL |
| イオン化モード | ESI Positive/Negative |
| イオンスプレー電圧 | 5500V/-4500V |
| イオンソース温度 | 350°C |
| 測定モード | sMRM (scheduled Multiple Reaction Monitoring) |



* Posのグラジエント: アセフェートなどの高極性農薬のピーク形状が改善

考察

液体試料だったが遠心分離後は、試料層が少しできた。

Positive測定農薬で全体的に回収率がやや高めに算出されたが、概ね良好な結果が得られた。一部の農薬は感度が悪い、またはRetention TimeがずれたのかStandardのピークが確認できず、分析不可のものがあった。

AISTI SCIENCE

Product

Smart-SPE C18-30
Smart-SPE C18-50
Smart-SPE PSA-30

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| No. | Pesticide Name | 添加回収率 | Spike回収率 | No. | Pesticide Name | 添加回収率 | Spike回収率 | No. | Pesticide Name | 添加回収率 | Spike回収率 |
|-----|------------------------|-------|----------|-----|-----------------------|-------|----------|-----------------|----------------------------|-------|----------|
| 1 | Abamectin | 85.4 | 102.0 | 81 | Hexaflumuron | 115.4 | 114.4 | 161 | Triflumuron | 127.6 | 125.4 |
| 2 | Acephate | 103.6 | 116.2 | 82 | hexazinone | 110.0 | 106.5 | 162 | Triticonazole | 84.8 | 90.4 |
| 3 | Acetamiprid | 120.0 | 108.7 | 83 | Hexythiazox | 96.4 | 106.8 | 163 | XMC | 117.8 | 125.9 |
| 4 | Acibenzolar-S-methyl | 91.7 | 105.5 | 84 | Imazalil | 110.5 | 127.0 | Negative | | | |
| 5 | Aldicarb | 108.6 | 116.4 | 85 | imazamethabenz-methyl | 111.3 | 111.9 | 1 | 2-4-D | 82.2 | 90.5 |
| 6 | Aldoxycarb | 82.4 | 86.6 | 86 | Imazaquin | 102.5 | 104.0 | 2 | 2-4-DP (Dichlorprop) | 94.5 | 99.9 |
| 7 | Anilofos | 104.5 | 107.7 | 87 | Imazosulfuron | 86.0 | 80.7 | 3 | 4-Chlorophenoxyacetic acid | 92.9 | 102.6 |
| 8 | Aramite | 131.0 | 139.3 | 88 | Imidacloprid | 104.6 | 99.0 | 4 | Acifluorfen | 96.0 | 99.3 |
| 9 | atrazine | 106.5 | 115.6 | 89 | Indanofan | 131.2 | 120.5 | 5 | Bromoxynil | 95.6 | 107.4 |
| 10 | Azamethiphos | 92.4 | 113.0 | 90 | Indoxacarb | 106.6 | 118.8 | 6 | Clopropr | 108.4 | 104.5 |
| 11 | Azimsulfuron | 88.9 | 103.4 | 91 | Iodosulfuron-methyl | 109.8 | 117.4 | 7 | Cyflanzilide | 103.2 | 106.9 |
| 12 | Azinphos-methyl | 105.1 | 113.8 | 92 | Iprodione | - | - | 8 | Dichlorprop | 94.5 | 99.9 |
| 13 | Azoxystrobin | 110.1 | 109.4 | 93 | Iprovalicarb | 109.4 | 113.3 | 9 | Fluroxypyr | 89.2 | 97.7 |
| 14 | Bendiocarb | 122.3 | 111.5 | 94 | Isoprocarb | 127.1 | 128.7 | 10 | Fomesafen | 96.7 | 111.3 |
| 15 | Bensulfuron-methyl | 101.6 | 109.1 | 95 | Isoxaflutole | 109.1 | 102.0 | 11 | Gibberellin | 97.9 | 109.3 |
| 16 | Benzofenap | 103.0 | 106.0 | 96 | Lactofen | 110.2 | 109.4 | 12 | Hexaflumuron | 100.2 | 109.9 |
| 17 | Boscalid | 111.2 | 104.7 | 97 | Lenacil | 99.9 | 108.5 | 13 | Ioxynil | 103.0 | 117.3 |
| 18 | bromacil | 82.4 | 82.4 | 98 | Linuron | 109.6 | 108.6 | 14 | Lufenuron | 100.4 | 104.6 |
| 19 | Butafenacil | 129.2 | 120.8 | 99 | Lufenuron | 94.3 | 87.6 | 15 | MCPA | 97.5 | 91.8 |
| 20 | Carbaryl | 100.6 | 104.3 | 100 | Mepanipyrim | 91.9 | 116.2 | 16 | MCPB | 98.8 | 103.5 |
| 21 | Carbofuran | 107.7 | 110.2 | 101 | Mesosulfuron-methyl | 91.5 | 102.4 | 17 | MCPB (Mecoprop) | 103.0 | 110.8 |
| 22 | carboxin | 85.6 | 102.5 | 102 | Methabenzthiazuron | 118.1 | 138.1 | 18 | Methoxyfenozide | 115.7 | 110.1 |
| 23 | Carpropamide | 98.3 | 109.6 | 103 | Methamidophos | 74.0 | 88.7 | 19 | Naphthaleneacetic acid | 92.3 | 91.4 |
| 24 | Chloridazon | 96.0 | 95.3 | 104 | Methiocarb | 121.5 | 115.4 | 20 | Naproanilide | 98.5 | 110.1 |
| 25 | Chlorimuron-ethyl | 110.4 | 102.0 | 105 | Methomyl | 168.3 | 118.5 | 21 | Oryzalin | 98.7 | 105.1 |
| 26 | Chlorsulfuron | 87.9 | 95.5 | 106 | Methoxyfenozide | 123.6 | 109.3 | 22 | Thidiazuron | 95.8 | 108.0 |
| 27 | Chlorxuron | 102.0 | 107.4 | 107 | Metosulam | 130.6 | 97.2 | 23 | Triclopyr | 126.1 | 100.5 |
| 28 | Chromafenozide | 115.8 | 124.6 | 108 | Metsulfuron-methyl | 107.2 | 133.0 | | | | |
| 29 | Cinosulfuron | 129.1 | 126.7 | 109 | mevinphosE | 117.9 | 120.1 | | | | |
| 30 | Clodinafop acid | - | - | 110 | mevinphosZ | 115.2 | 136.3 | | | | |
| 31 | Clofencet | 41.8 | 41.7 | 111 | monocrotophos | 106.1 | 103.9 | | | | |
| 32 | Clofentezine | 121.6 | 108.7 | 112 | Monolinuron | 96.7 | 86.3 | | | | |
| 33 | Clomeprop | 106.4 | 123.3 | 113 | Naproanilide | 96.2 | 108.7 | | | | |
| 34 | Cloquintocet-mexyl | 102.0 | 110.1 | 114 | Naptalam | 71.8 | 84.6 | | | | |
| 35 | Cloransulam-methyl | 93.7 | 98.7 | 115 | Novaluron | 107.2 | 121.9 | | | | |
| 36 | Clothianidin | 117.4 | 137.8 | 116 | oxadixyl | 107.8 | 82.9 | | | | |
| 37 | Cumyruron | 117.8 | 114.1 | 117 | Oxamyl | 99.8 | 94.0 | | | | |
| 38 | cyanazine | 125.9 | 121.4 | 118 | Oxaziclonofone | 109.5 | 110.5 | | | | |
| 39 | Cycloate | 88.6 | 126.9 | 119 | Oxycarboxin | 103.6 | 109.1 | | | | |
| 40 | Cyclosulfamuron | 115.3 | 117.7 | 120 | Pencycuron | 96.2 | 109.8 | | | | |
| 41 | Cyflufenamide | 115.7 | 117.3 | 121 | Penoxsulam | 113.2 | 111.4 | | | | |
| 42 | Cyprodinil | 104.1 | 116.1 | 122 | Phenmedipham | 106.2 | 109.4 | | | | |
| 43 | DDVP | 107.5 | 103.6 | 123 | PhosphamidoneE | 115.9 | 109.6 | | | | |
| 44 | Di-allate | 130.1 | 107.2 | 124 | PhosphamidoneZ | 91.2 | 75.9 | | | | |
| 45 | Diazinon-d | - | - | 125 | Primicarb | 92.5 | 108.8 | | | | |
| 46 | Dichlosulam | 139.9 | 135.6 | 126 | Primicarb | 97.0 | 108.5 | | | | |
| 47 | Diclorfentazine | 121.1 | 104.2 | 127 | Propaquizafop | 118.1 | 127.0 | | | | |
| 48 | Diclotopos | 100.3 | 100.8 | 128 | propoxur | 124.6 | 123.8 | | | | |
| 49 | Diflubenzuron | 88.9 | 88.6 | 129 | Propoxycarbazono | 105.0 | 166.6 | | | | |
| 50 | Dimethirimol | 110.5 | 100.0 | 130 | Pyraclostrobin | 96.2 | 115.0 | | | | |
| 51 | Dimethoate | 120.1 | 118.8 | 131 | Pyrazolynate | 106.1 | 112.4 | | | | |
| 52 | DimethomorphE | 91.6 | 122.0 | 132 | Pyrazosulfuron-ethyl | 104.2 | 108.2 | | | | |
| 53 | DimethomorphZ | 125.5 | 108.3 | 133 | Pyriflitalid | 91.9 | 103.9 | | | | |
| 54 | Dimeton-s-methyl | 119.2 | 120.9 | 134 | pyroquilon | 102.4 | 107.6 | | | | |
| 55 | Diuron | 123.8 | 123.0 | 135 | Quizalofop-ethyl | 96.9 | 114.8 | | | | |
| 56 | Dymuron | 115.6 | 125.7 | 136 | Quizalofop-P-tefuryl | - | - | | | | |
| 57 | Epoxiconazole | 117.1 | 104.2 | 137 | Silafluofen | 21.5 | 97.4 | | | | |
| 58 | Ethametsulfuron-methyl | 109.8 | 106.1 | 138 | Simazine | 101.6 | 89.8 | | | | |
| 59 | Ethoxysulfuron | - | - | 139 | Simeconazole | 100.6 | 105.1 | | | | |
| 60 | Fenamidone | 104.2 | 107.6 | 140 | Simetorin | 108.7 | 112.2 | | | | |
| 61 | Fenhexamid | 84.9 | 91.8 | 141 | Spinosyn A | 86.1 | 92.4 | | | | |
| 62 | Fenobucarb | 95.0 | 102.6 | 142 | Spinosyn D | 82.8 | 99.0 | | | | |
| 63 | Fenoxaprop-ethyl | 97.1 | 60.0 | 143 | Sulfentrazone | 94.3 | 96.8 | | | | |
| 64 | Fenoxycarb | 107.7 | 113.9 | 144 | Sulfosulfuron | 91.0 | 130.8 | | | | |
| 65 | Fenpyroximate E | 94.2 | 109.1 | 145 | Tebufenozide | 103.0 | 96.6 | | | | |
| 66 | Fenpyroximate Z | 92.6 | 105.1 | 146 | Tebuthiuron | 94.9 | 103.9 | | | | |
| 67 | Ferimzone EandZ | 105.8 | 115.3 | 147 | Teflubenzuron | 115.1 | 116.8 | | | | |
| 68 | Flazasulfuron | 100.6 | 109.9 | 148 | terbacil | - | - | | | | |
| 69 | Florasulam | 118.9 | 119.9 | 149 | Tetrachlorvinphos | 107.0 | 120.4 | | | | |
| 70 | Fluazifop | 127.6 | 117.1 | 150 | Thiabendazole | 88.4 | 104.1 | | | | |
| 71 | Flufenacet | 119.2 | 113.3 | 151 | Thiacloprid | 110.2 | 110.4 | | | | |
| 72 | Flufenoxuron | 94.7 | 105.2 | 152 | Thiamethoxam | 96.4 | 115.2 | | | | |
| 73 | Flumetsulam | 115.9 | 125.9 | 153 | Thidiazuron | 102.2 | 125.6 | | | | |
| 74 | Fluridon | 115.0 | 101.4 | 154 | Thifensulfuron-methyl | 110.9 | 127.8 | | | | |
| 75 | Foramsulfuron | 94.2 | 105.1 | 155 | Thiodicarb | 43.5 | 111.7 | | | | |
| 76 | Forchlorfenuron | 103.9 | 117.8 | 156 | Tralkoxydim | 102.3 | 124.6 | | | | |
| 77 | Furametpyr | 106.4 | 115.0 | 157 | Triasulfuron | 137.3 | 114.0 | | | | |
| 78 | Furathiocarb | 109.9 | 115.8 | 158 | Tridemorph E | 106.4 | 107.1 | | | | |
| 79 | Halosulfuron-methyl | 143.6 | 107.8 | 159 | Tridemorph Z | 104.1 | 107.9 | | | | |
| 80 | Haloxyfop | 106.6 | 108.9 | 160 | Trifloxysulfuron | 116.6 | 99.8 | | | | |

* Spike回収率：最終試験溶液に農薬を添加し測定することで、機器分析におけるマトリックス効果を確認。

* 絶対検量線を使用 回収率30%未満は スタンダードピークなしは 回収率150%以上は