# GC-MS Investigation of Tropane Alkaloids in *Datura stramonium*

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The alkaloid spectrum in roots, leaves and seeds of *Datura stramonium* L. was investigated by GC-MS. Twenty-nine tropane alkaloids are detected. Twelve of them are new constituents for the species and the two tropane esters 3-(3′-acetoxytropoyloxy)tropane (**21**) and 3-(2′-hydroxytropoyloxy)tropane (**26**) are described for the first time.

GC-MS is useful method for separation and identification of complex mixtures of tropane alkaloids (Witte *et al*., 1987; Ionkova *et al*., 1994). To our knowledge, there are scanty data for the alkaloid spectrum of *Datura stramonium* L. Twenty-eight tropane alkaloids are reported for the species until now and nineteen of them for intact plants (Parr *et al*., 1990; Robins *et al*., 1991; Lounasmaa and Taminnen, 1993; Ionkova *et al*., 2000; Berkov and Philipov, 2001).

Twenty-nine alkaloids from the extracts of *D. stramonium* (Bulgarian origin) have been detected by GC-MS (Table I). They all have the characteristics for the tropane nucleus ions at *m/z* 124, 113, 96, 95, 94, 83, 82 and 81 (Blossey *et al*., 1964). To our knowledge twelve of the alkaloids identified are new for this species. Alkaloids A, B and C were left unidentified. Alkaloids **3** and **4**, **9** and **10**, **18** and **19**, **24** and **25** appear as double peaks in GC-MS with identical mass spectra. They are isomeric tropine and pseudotropine esters (*Witte et al*., 1987). The isomeric forms of the mentioned tropane esters are new constituents in the alkaloid spectrum of *D. stramonium.* The stereochemistry of **3**, **4**, **9**, **10**, **18**, **19**, **24** and **25** was not discussed as it could not be established solely by MS data.

Alkaloid **21** has $M^+$ at *m/z* 331. The base peak at *m/z* 124 together with the ion at *m/z* 140 ($M^+-191$) suggests a C-3 substitution. Fragments at *m/z* 288 ($M^+-43$; $C_2H_3O$), *m/z* 272 ($M^+-59$; $C_2H_3O_2$), *m/z* 207 ($C_{11}H_{11}O_4$) and *m/z* 43 ($C_2H_3O$) indicate an acetyltropoyl ester group at C-3. On the basis of its mass spectral fragmentation alkaloid **21** was identified as 3-(3′-acetoxytropoyloxy)tropane – a new tropane alkaloid (Fig. 1).

Alkaloid **26** shows $M^+$ at *m/z* 305 and base peak at *m/z* 124. The ion at *m/z* 140 ($M^+-165$) suggests a group with 165 mass units at C-3. From the known moieties of the tropanes only a 2-hydroxytropoyl group has 165 mass units. Such a moiety is observed at C-3 in 3-(2′-hydroxy)tropoyloxy-6,7-epoxytropane (daturamine), which is an alkaloid distributed in the genera *Datura*, *Scopolia* and *Przewalskia* (Lounasmaa and Taminnen, 1993). On the basis of these data the structure of alkaloid **26** was elucidated as 3-(2′-hydroxytropoyloxy)tropane – a new tropane alkaloid (Fig. 1).

## Material and Methods

The seeds of *D. stramonium* were collected from natural habitats in the vicinities of Lovech, Bulgaria. A voucher specimen No CO-529 (SOM) was deposited at the Herbarium of the Institute of

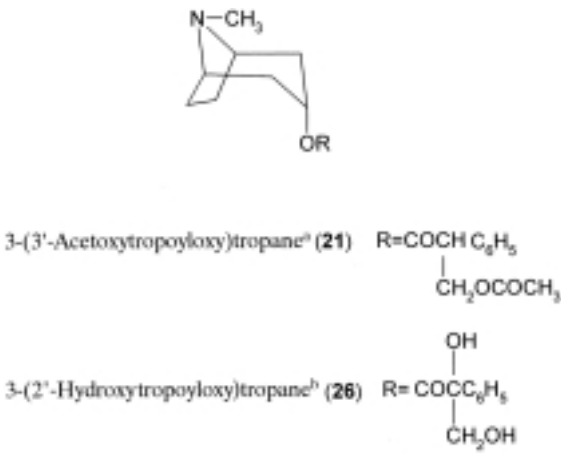


Fig. 1.

  

Table I. Alkaloids found in *D. stramonium*.

| Compounds | $M^+$and base peak | Roots | Leaves | Seeds | MS Ref. | Compound reported to occur in *D. stramonium* |
|---|---|---|---|---|---|---|
| 3-Acetoxy-6-hydroxytropane (**1**) | 199, 94 | + | – | – | (2) | – |
| 3-Tygloyloxytropane (**2**) | 223, 124 | + | – | + | (1, 2) | (3) |
| 3α(?)-Phenylacetoxytropane (**3**) | 259, 124 | – | – | + | (2) | (6[c]) |
| 3β(?)-Phenylacetoxytropane (**4**) | 259, 124 | – | – | + | (2) | – |
| 3- Tygloyloxy-6,7-dihydroxytropane (**5**) | 255, 94 | + | – | – | (1, 2) | (3, 10) |
| 3-Hydroxy-6-tygloyloxytropane (**6**) | 239, 113 | + | – | – | (1) | – |
| 3-Tygloyloxy-6-hydroxytropane (**7**) | 239, 94 | + | – | – | (1) | (3[c], 4[c]) |
| 3-Tygloyloxy-6-isobutyryloxytropane (**8**) | 309, 94 | + | – | – | (1) | – |
| 3α(?)-Apotropoyloxytropane (**9**) | 271, 124 | + | + | + | (1, 2) | (3, 10) |
| 3β(?)-Apotropoyloxytropane (**10**) | 271, 124 | – | – | + | (1, 2) | – |
| 3-Tygloyloxy-6-methylbutyryloxytropane (**11**) | 323, 94 | + | – | – | (1) | – |
| Alkaloid À (**12**) | –, 124 | + | – | – | – | – |
| Alkaloid 325 (**13**) | 325, 94 | + | – | – | (4) | (4[c]) |
| Alkaloid B (**14**) | –, 94 | + | – | – | – | – |
| 3-Apotropoyloxy-6,7-epoxytropane (**15**) | 285, 94 | – | – | + | (1) | (3) |
| 3-Tropoyloxytropane (**16**) | 289, 124 | + | + | + | (1, 2) | (3, 10) |
| 3,6-Ditygloyloxytropane (**17**) | 321, 94 | + | – | – | (1, 2) | (3, 10) |
| 3α(?)-Tygloyloxy-6-isovaleroyloxy-7-hydroxytropane (**18**) | 339, 94 | + | – | – | (7, 9) | (7) |
| 3β(?)-Tygloyloxy-6-isovaleroyloxy-7-hydroxytropane (**19**) | 339, 94 | + | – | – | (7, 9) | (7) |
| Methylscopolamine (**20**) | 317, 94 | – | + | – | (2) | – |
| 3-(3′Acetoxytropoyloxy)tropane [a] (**21**) | 331, 124 | + | – | – | – | – |
| 3-Tropoyloxy-6,7-epoxynortropane (**22**) | 289, 122 | + | – | – | (2) | (3) |
| 3-Tropoyloxy-6,7-epoxytropane (**23**) | 303, 94 | + | + | + | (1, 2) | (3, 10) |
| 3α(?),6β-Ditygloyloxy-7β-hydroxytropane (**24**) | 337, 94 | + | – | – | (1, 2) | (3, 10) |
| 3β(?),6β-Ditygloyloxy-7β-hydroxytropane (**25**) | 337, 94 | + | – | – | (1, 2) | – |
| 3-(2′-Hydroxytropoyloxy)tropane [b] (**26**) | 305, 124 | + | – | – | – | – |
| 3-Tropoyloxy-6-hydroxytropane (**27**) | 305, 94 | + | – | – | (2) | – |
| 3-Tropoyloxy-6-tygloyloxytropane (**28**) | 387, 94 | + | – | – | (1, 2) | – |
| Alkaloid C (**29**) | –, 124 | + | – | – | – | – |

*m/z* (rel. int.%):
[a] $M^+$ 331 (6), 288 (1), 272 (2), 140 (5), 124 (100), 96 (8), 94 (18), 82 (17), 67 (8), 43 (12).
[b] $M^+$ 305 (4), 288 (1), 140 (8), 124 (100), 94 (20), 83 (25), 55 (22).
[c] In *in vitro* cultures

Botany, Bulgarian Academy of Sciences. The plants were grown at controlled condition as reported by Berkov and Philipov, (2001). In the first flower phase, the leaves and roots of plants were collected and dried at 45 °C. Alkaloid extraction and GC-MS measurements were made after Berkov and Philipov, (2001). The identification of alkaloids was done by comparison with literature data (Evans and Major, 1968; Witte *et al.,* 1987; Ionkova *et al.,* 1994) as well as with the Wiley data base 275 and NIST 1998.

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