Zagazig J. Agric. Res., Vol. 35 No. (6) 2008 1415-1422

ISOLATION AND IDENTIFICATION OF TWO NEW COMPOUNDS FROM *SOLENOSTEMMA ARGEL* HAYNE (FAM. *ASCLEPIADACEAE*)

Shalash, Eman. M., M.M.A. Amer, A.M. Abo-Eyta and S.A. Gaber

Agricultural Biochemistry Department, Faculty of Agriculture, Zagazig University.

Accepted 30/11/2008

ABSTRACT: The hexane extract of *Solenostemma argel* Hayne (Fam. *Asclepiadaceae*), yielded two new compounds identified by spectroscopical methods as heneicosanyl pentanoate and lupeol- 3-methyl ketone. The structures of the isolated compounds were verified by means of melting point, IR and Mass spectral analysis.

Key words:*Solenostemma argel* Hayne, Asclepiadaceae, Heneicosanyl pentanoate, Lupeol-3-methyl ketone, Melting point, IR, Mass spectral analysis.

INTRODUCTION

Solenostemma argel Hayne (Fam. *Asclepiadaceae*) is an Egyptian wild perennial erect shrub growing in the eastern desert and alongside the Nile banks in South Egypt (Boulos, 2000). It is the only species found in Egypt from the genus solenostomma (El-Hadidi *et al.*, 1994). The leaves are commonly used in traditional medicine as a purgative, antipyretic, expectorant, antispasmodic and in case of bile congestion (Hocking, 1955). Previous studies have reported the occurrence of two monoterpene glucosides (Kamel *et al.*, 2000).

Michael (1998) isolated flavonol diglycoside. Also, Hamed (2001) isolated two new pregnane ester glycosides, named stemmoside A and stemmoside B. Hassan *et al.* (2001) isolated two new pregnane derivatives, in addition to α- and β-amyrin and β-sitositrol. Kamel (2003) isolated four new acylated phenolic glycosides Solargin I, Solargin II, Solargin III and Solargin IV. Furthermore, Plaza *et al.* (2004) isolated 14, 15- Keto- pregnane glycosides from the pericarps of *Solenostemma argel*. The same authors (2005) isolated eight new

14, 15- secopregnane glycosides from the pericarps of *Solenostemma argel.* In addition, Perrone *et al.* (2006) isolated five new 14, 15-secopregnane derivatives, named argelosides K-O from *Solenostemma argel.*

MATERIALS AND METHODS

Plant Material

The aerial parts of *Solenostemma argel* Heyne (Fam. *Asclepidaceae*) were collected in May 2003 from North Sinai and identified by Faculty of Science herbarium, Cairo University. The plant material was dried in shade and grounded to moderately fine powder.

Herbarium specimen was kept at the Department of Agricultural Biochemistry, Faculty of Agriculture, Zagazig University, at 25°C until analysis.

Extraction and Isolation

The air- dried powdered aerial parts of *Solenostemma argel* Hayne (1.5Kg) were extracted by steeping in hexane (3Lx6). The pooled hexane extract was evaporated under vacuum at 50°C till dryness. About 5.0g of the hexane extract was dissolved in a small amount of chloroform then mixed with 5.0g of silica gel and evaporated under vacuum at about 50°C till dryness. The obtained dried powder was applied to the top of a glass column (30x3cm) packed with 100g of silica gel (200-400 mech, S.D. Fine- chem. Limited, Mumbai, India) in petroleum ether. Gradient elution was started with petroleum ether, followed by gradual increase of chloroform (95: 5, 90: 10, 85: 15, 80: 20, ..., 5: 95 and 100% chloroform). The collected fractions were examined on TLC slides and similar fractions were pooled together.

Spray Reagents

1. P-Anisaldehyde- Sulphuric acid spray reagent (Wagner *et al.*, 1984) for compound 1.
2. Liebermann- Burchard test (Liebermann and Burchard, 1890) for compound 2.

General

- Hexane
- Petroleum ether (60-80°C)
- Chloroform
- P- Anisaldehyde – Sulphuric acid spray reagent
- Acetic anhydride – Sulphuric acid reagent.
- precoated TLC plates silica gel 60 GF^{254} (E.Merck) (20x20 cm).

- Rotatory evaporator (Buchi).
-The melting points were measured using Electrothermal AZ 9003
- Jasco 460 plus FT/IR, made in Japan, Infra red spectrophotometer.
- The mass spectra were carried out on a Finnigan MATSSQ 700 system in EL mode scan.

RESULTS AND DISCUSSION

Compound 1

Compound 1 occuring as white powder, with m.p 95°C, showed a single spot with R_f 0.36 on TLC using solvent system (Petroleum ether: chloroform, 9:1) then visualized with anisaldehyde sulphuric acid spray reagent. This compound is soluble in petroleum ether, insoluble in ethanol and methanol.

This compound showed infrared spectrum with strong and sharp absorption peaks at 2918 and 2849 cm^{-1} indicating the presence of large number of CH, CH_2 and CH_3 stretching vibration; sharp absorption frequency at 1734 cm^{-1} indicating the presence of carbonyl group of ester; absorption frequency at 1467 cm^{-1} and 1416 cm^{-1} together with peaks at 1375 cm^{-1} indicating the presence of large number of C-C aliphatic stretching vibration and supported with absorption frequencies at 1176 cm^{-1} and 1043 cm^{-1} characteristic for C-O stretching of the carbonyl. This infrared absorptions peaks suggest that the compound is a long chain hydrocarbon with an ester group (Yamaguchi, 1970).

The mass spectrum showed molecular ion peak (M^+) at m/z 396 (7%) which corresponds to a molecular formula C_{26} H_{52} O_2, and fragment ion peaks at m/z 397 (16%, M^+ +1), m/z 368 (11%, M^+ - 28) suggesting the loss of a carbonyl group (C=O), m/z 339 (2%, M^+ -57 or M^+ - C_4H_9) 352 (2%, M^+- 44), m/z 340 (13%), 312 (19%, 340-28), 284 (2%, 312-28), 256 (2%, 284-28) suggesting successive losses of ethylene (C_2H_4). In addition, m/z 57 (100%), 71 (56%, 57-14), 85 (36%, 71-14) and 99 (9%, 85-14) suggesting scuccessive losses of CH_2.

The placement of the carbonyl group was confirmed by the peaks at m/z 57 (100%) for C4 unit at one side of the carbonyl and m/z 339 (2%) for ($C_{21}HO_2$) and 340 (13%) for m/z 339 + H and m/z

341 (41%) (340 + H) (Yamaguchi, 1970).

The above data confirmed that this compound is heneicosanyl pentanoate as shown in Fig 1.This is the first time of isolation of this compound from the plant.

Compound 2

The second identified compound was obtained as white scales, with m.p 110C°. This compound is soluble in chloroform, insoluble in methanol and ethanol. It gave blue colour with Liebermann – Burchard test for sterol or triterpen. (Liebermann – Burchard, 1890)

This compound has shown infrared spectrum with strong and sharp absorption peaks at 2918 and 2849 cm^{-1} indicating the presence of large number of C-H stretching vibration; sharp absorption frequency at 1705 cm^{-1} indicating the presence of carbonyl group C=O; absorption frequency at 1560 cm^{-1} indicating the presence of C=C stretching; absorption frequency at 1467 cm^{-1} and 1433 cm^{-1} together with peaks at 1376 cm^{-1} indicated the presence of larg number of C-C aliphatic stretching vibration. These data confirm the presence of a steroid or triterpens (Goad and Akihisa, 1997). The mass spectrum showed a molecular ion peak (M^+) at m/z 452 (99%) which corresponds to a molecular formula C_{32} H_{52} O, and fragment ion peaks m/z 437 (13%, M^+- CH_3 or M^+ - 15), m/z 4 (49%, M^+ - 28 or M^+ - CO or M^+ - C_2H_4), m/z 409 (19%, M^+ - 43 or M^+ - CO – CH_3) confirming the presence of methyl keton. These data with typical fragmentation for compounds with this class of pentacyclic triterpens (Goad and Akihisa, 1997). The above data suggested that this compound is lupeol - 3- methyl ketone as shown in Fig 2. This is the first report on this compound from the plant.

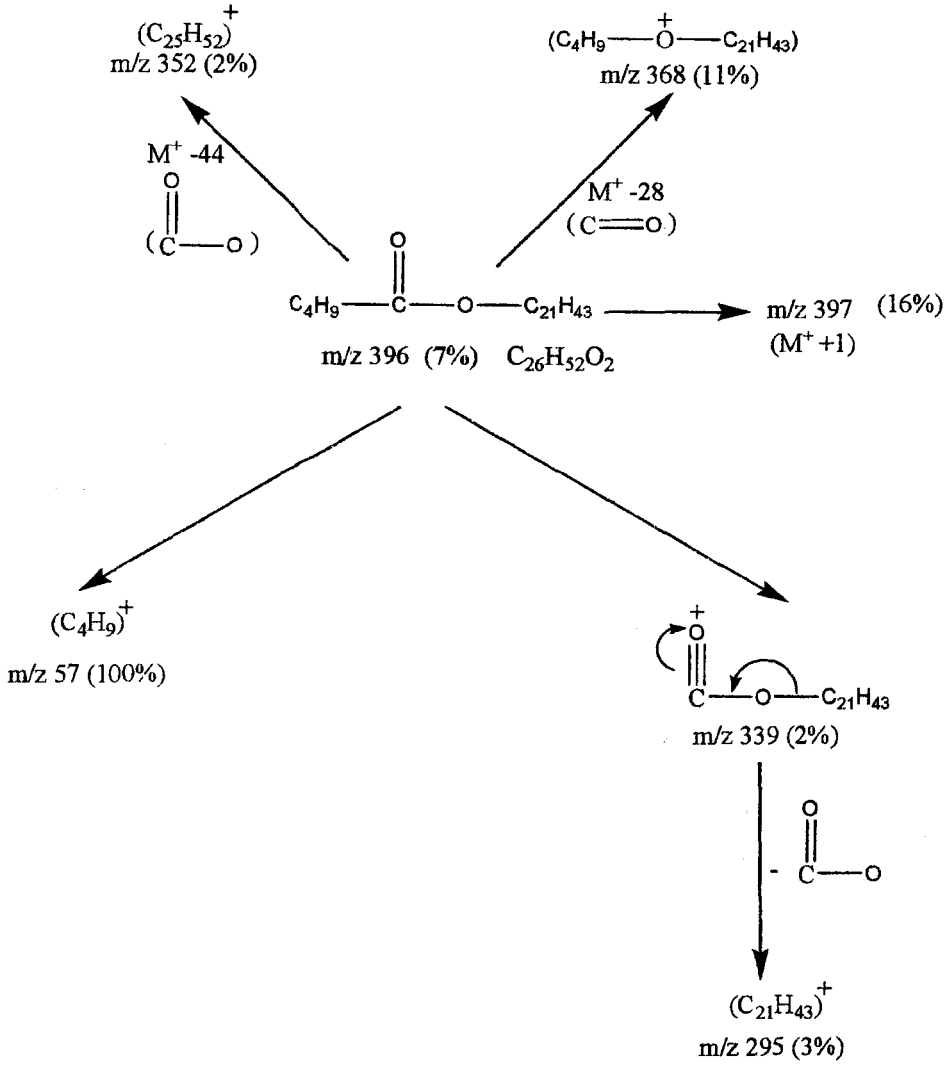

Fig. 1. Fragmentation of compound1

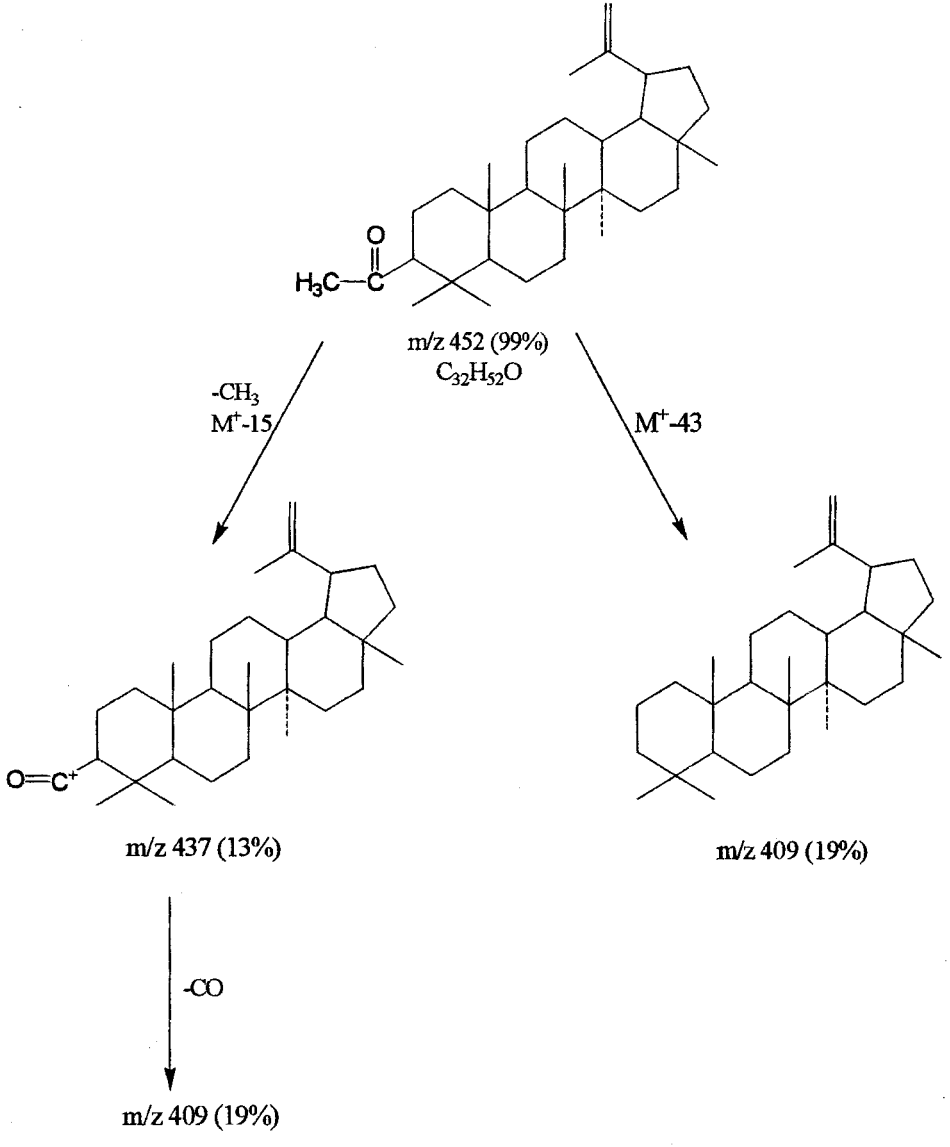

Fig. 2. Fragmentation of compound2

REFERENCES

Boulos, L. 2000. Flora of Egypt (volume two), Ed. Al hadara pubishing.

El-Hadidi, M.N. and F. Abdel-Aziz. 1994. Tackholmia, materials for Excursion Flora of Egypt (EFE) Vol. 15. El-Arabi Printhouse. Cairo 118.

Goad, L.J. and T. Akihisa. 1997. Analysis of Sterols. 1st Ed. T. J, press, chapman and Hall Landon.

Hocking, G. 1955. A dictionary of Terms in pharmacognosy and Other Division of Economic Botany. Charles Thomas Publisher, Oxford. P. 212.

Hamed, A. 2001. New Steroids from *Solenostemma argel* leaves. Fitoterapia. 72(7), 747-755.

Hassan, H.A.; A.I. Hamed; N.A. I-Emary; I.V. Springuel; H. Mitome and H. Miyaoka. 2001. pregnane derivatives from *Solenostemma argel* leaves. Phytochemistry. 57, 507-511.

Kamel, M.S.; K. Ohtani; H.A. Hassanain; M.H. Mohamed; R. Kasai and K. Yamasaki. 2000. Monoterpene and preganane glucosides from *Solenostemma argel.* Phytochemistry. 53 (8), 937-940.

Kamel, M.S. 2003. Acylated phenolic glycosides from *Solenostemma argel.* Phytochemistry. 62, 1247-1250.

Liebermann, C. and H. Burchard. 1890. Chem. Zentr. 61.7.

Michael, Helana. N. 1998. A new flavonol diglycoside from *Solenostemma argel* leaves. Asian. J. Chem. 10 (4), 1038-1040.

Plaza, A.; S. Piacente; A. Perrone; A. Hamed; C. Pizza and G. Bifulco. 2004. Stemmosides C and D, two novel unusual pregnane glycosides from *Solenostemma argel*: structural elucidation and configurational stady by a combined NMR-quantum mechanical strategy. Tetrahedron. 60 (52), 12201-12209.

Plaza, A.; A. Perrone; C. Balestrieri; M.L. Balestrieri; G. Bifulco, V. Carbone; A. Hamed; C. Pizza and S. Piacente. 2005. New antiproliferative 14,15-secopregnane glycosides from *Solenostemma argel.* Tetrahedron. 61 (31), 7470-7480.

Perrone, A.; A. Plaza; S.F. Ercolino; A.I. Hamed; L. Parente; C. Pizza and S. Piacente. 2006. 14, 15-secopregnane derivatives from the leaves of *Solenostemma argel*. Journal of Natural Products. 69 (1), 50-54.

Wagner, H.; S. Bladt and E.M. Zgainski 1984. Plant Drug Analysis, Springer Verlag, Berlin.

Yamaguchi, K. 1970. Spectral Data of Natural Products. Vol. I. Elsevier publishing company, Amsterdam, London and New York.

فصل مركبين جديدين من نبات الحرجل والتعرف عليهما

إيمان محمد شلش- محمد مصطفى عفيفى عامر-

أحمد محمد أبو عيطة – سعيد عبد الحميد جبر

قسم الكيمياء الحيوية الزراعية- كلية الزراعة- جامعة الزقازيق

تم التعرف على مركبين من مستخلص الهكسان لنبات الحرجل وهما هين إيكوزانول بنتا نوات وليبيول-٣- ميثيل كيتون، وقد تم التعرف على المركبات المفصولة باستخدام تحليل طيف الأشعة تحت الحمراء و مقياس طيف الكتلة ودرجة الانصهار.