

*Preparation of methyl esters of fatty acids.* Lipid samples ca 10 mg were methylated [16] in a special extraction flask. Methyl esters were analysed by GC at 200° on a 2 m glass column packed with 6% diethylene glycol succinate on diatoport S with N<sub>2</sub> as carrier. The peaks were identified by comparison with known standards. Percentages of individual fatty acids were calculated by triangulation.

*Acknowledgements*—We are grateful to Professor P. S. Sastry, Professor of Biochemistry, Indian Institute of Science, Bangalore for GC facilities. Thanks are due to Professor V. S. R. Das for providing laboratory facilities. G.R.G. is in receipt of a fellowship from CSIR, New Delhi.

#### REFERENCES

1. Worthington, R. E. and Hammons, R. O. (1971) *Oleagineux* **26**, 695.
2. Worthington, R. E., Hammons, R. O. and Allison, J. R. (1972) *J. Agric. Food Chem.* **20**, 727.
3. Har-tzook, A. (1969) *Curr. Sci.* **38**, 176.
4. Young, C. T., Mason, M. E., Matlock, R. S. and Waller, G. R. (1972) *J. Am. Chem. Soc.* **49**, 314.
5. Kuiper, P. J. C. in *Biogenesis and Function of Plant Lipids* (1980) (Mazliak, P., Benveniste, P., Costes, C. and Douce, R., eds.). Elsevier/North-Holland, Amsterdam.
6. Lyons, J. M. (1973) *Annu. Rev. Plant Physiol.* **24**, 445.
7. Willemot, C. (1977) *Plant Physiol.* **60**, 1.
8. Kuiper, P. J. C. (1970) *Plant Physiol.* **45**, 684.
9. Smolenska, G. and Kuiper, P. J. C. (1977) *Physiol. Plant.* **41**, 29.
10. John, J. B. St. and Christensen, M. N. (1976) *Plant Physiol.* **57**, 257.
11. Vyas, D. N., Patel, K. C. and Patel, R. D. (1969) *J. Am. Oil Chem. Soc.* **46**, 41.
12. Stuver, C. E. E., Kuiper, P. J. C. and Marschner, H. (1978) *Physiol. Plant* **42**, 124.
13. Scarpa, A. O. and de Gier, J. (1971) *Biochim. Biophys. Acta.* **241**, 789.
14. Alfrey, J. M. and Northcote, D. H. (1977) *New Phytol.* **78**, 547.
15. Folch, J., Lees, M. and Stanley, G. H. (1957) *J. Biol. Chem.* **226**, 497.
16. Kates, M. (1964) *J. Lipid Res.* **5**, 132.

*Phytochemistry*, Vol. 21, No. 11, pp. 2742–2743, 1982.  
Printed in Great Britain.

0031-9422/82/112742-02\$03.00/0  
© 1982 Pergamon Press Ltd.

## METHYL HEXADECA-6,8,12-TRIEN-10-YNOATE FROM *CHRYSOCOMA TENUIFOLIA*\*

FERDINAND BOHLMANN and MANIRUDDIN AHMED

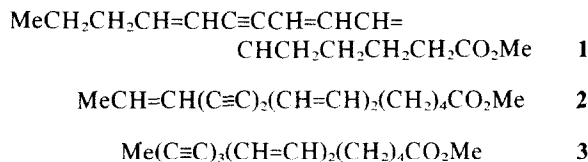
Institute for Organic Chemistry, Technical University of Berlin, D-1000 Berlin 12, West Germany

(Received 22 February 1982)

**Key Word Index**—*Chrysocoma tenuifolia*; Compositae; acetylenic compound; methyl hexadeca-6,8,12-trien-10-ynoate.

**Abstract**—*Chrysocoma tenuifolia* afforded a new C<sub>16</sub>-acetylenic ester.

*Chrysocoma* is a South African genus belonging to the tribe Astereae. Three species have been investigated previously [1], two of them afforded acetylenic esters with an unusual C<sub>16</sub>-chain. A reinvestigation of *C. tenuifolia* Berg., collected in Transvaal, only gave one such ester, the enynediene, **1**, as followed from the spectroscopic data. While from the UV maxima the nature of the chromophore could be deduced, the molecular formula clearly showed the presence of a C<sub>16</sub>-ester. The positions of the double



bonds, however, could not be indicated from the fragmentation pattern. The <sup>1</sup>H NMR spectrum (Table 1) clearly showed that all three double bonds were *trans*-configured, while the position of the double bonds could be assigned by careful spin decoupling. Starting with the signal of the terminal methyl group the signal of H-15 could be assigned. Irradiation of

\*Part 263 in the series "Polyacetylenic Compounds". For Part 262 see Bohlmann, F., Ahmed, M., King, R. M. and Robinson, H., *Phytochemistry* (in press).

Table 1.  $^1\text{H}$  NMR spectral data of compound **1** (400 MHz, TMS as internal standard)

	$\text{C}_6\text{D}_6$	$\text{CDCl}_3$
H-2	2.07 t	2.31 t
H-3	1.47 tt	1.61 tt
H-4	1.12 tt	1.41 m
H-5	1.81 dt	2.08 dt
H-6	5.43 dt	5.74 dt
H-7	5.88 dd	6.06 dd
H-8	6.79 dd	6.51 dd
H-9	5.74 br d	5.58 br d
H-12	5.70 br d	
H-13	6.18 dt	6.11 dt
H-14	1.83 dt	2.11 dt
H-15	1.16 tq	1.41 m
H-16	0.74 t	0.88 t
OMe	3.38 s	3.66 s

$J$  (Hz): 2, 3 = 3, 4 = 4, 5 = 5, 6 = 13, 14 = 14, 15 = 15, 16 ~ 7; 6, 7 = 8, 9 = 12, 13 = 15; 7, 8 = 10.

the latter collapsed the broadened double triplet to a doublet. As this signal was further coupled with the doublet triplet at  $\delta$  6.18, which itself was coupled with a proton adjacent to the triple bond, the positions of the double bonds were established. Similarly the

signals of H-2–H-9 could also be assigned. Since the acetylenic ester **2** and **3** had been isolated previously from the same plant, the question arose whether **1** is the precursor of **2** and **3** or formed by hydrogenation of **3** via **2**. So far this point of general interest in the biogenesis of acetylenic compounds is not established though most of these steps were confirmed by feeding experiments [2].

#### EXPERIMENTAL

The air-dried aerial parts (30 g) (voucher 81/216, deposited in the Botanic Research Institute, Pretoria) were extracted with  $\text{Et}_2\text{O}$ –petrol (1:2) and the resulting extract was separated by TLC (Si gel) using  $\text{Et}_2\text{O}$ –petrol (1:10). 4 mg **1** were obtained, colourless gum,  $\text{IR}_{\text{max}}^{\text{CCl}_4} \text{ cm}^{-1}$ : 2180 (very weak,  $\text{C}\equiv\text{C}$ ), 1740 ( $\text{CO}_2\text{R}$ ), 1640, 990, 960 [*trans*, *trans* ( $\text{CH}=\text{CH})_2$  and *trans*  $\text{CH}=\text{CH}$ ]; UV  $\lambda_{\text{max}}^{\text{Et}_2\text{O}}$  nm: 310, 290. MS  $m/z$  (rel. int.): 260.178  $[\text{M}]^+$  (28 ( $\text{C}_{17}\text{H}_{24}\text{O}_2$ ), 229  $[\text{M} - \text{OMe}]^+$  (2), 217  $[\text{M} - \text{C}_3\text{H}_7]^+$  (4), 157  $[\text{C}_{12}\text{H}_{13}]^+$  (21), 143  $[\text{C}_{11}\text{H}_{11}]^+$  (21), 129  $[\text{C}_{10}\text{H}_9]^+$  (44), 117  $[\text{C}_9\text{H}_7]^+$  (100), 91  $[\text{C}_7\text{H}_7]^+$  (58).

**Acknowledgements**—We thank Dr. B. de Winter and Miss M. Welman, Botanic Research Institute, Pretoria, for their help and the Deutsche Forschungsgemeinschaft for financial support.

#### REFERENCES

1. Bohlmann, F. and Zdero, C. (1972) *Chem. Ber.* **105**, 3587.
2. Bohlmann, F., Burkhardt, T. and Zdero, C. (1973) *Naturally Occurring Acetylenes*. Academic Press, London.

*Phytochemistry*, Vol. 21, No. 11, pp. 2743–2745, 1982.  
Printed in Great Britain.

0031-9422/82/112743-03\$03.00/0  
© 1982 Pergamon Press Ltd.

## SESQUITERPENE LACTONES FROM *INEZIA INTEGRIFOLIA*\*

FERDINAND BOHLMANN and CHRISTA ZDERO

Institute for Organic Chemistry, Technical University of Berlin, D-1000 Berlin 12, West Germany

(Received 21 December 1981)

**Key Word Index**—*Inezia integrifolia*; Compositae; sesquiterpene lactones; eudesmanolides; guaianolide.

**Abstract**—*Inezia integrifolia* afforded, in addition to known compounds, three new eudesmanolides and a guaianolide. Two of the eudesmanolides were obtained as a mixture. The relative position of the ester groups could not be determined with certainty. The chemotaxonomy is discussed briefly.

*Inezia* is a monotypic genus placed in the tribe Anthemideae. Morphological data indicated a relationship to *Lidbeckia* and *Thaminophyllum* [1], all

three genera being endemic in South Africa. As the chemistry of the latter two genera has been studied [2, 3], it was of interest whether that of *Inezia* supports the proposed relationship. The roots of *I. integrifolia* (Klatt.) Phill. afforded **1** and **2** [4] and nerol isovalerate (**3**), while the aerial parts gave germacrene D, bicyclogermacrene, linoleic and linolenic acids as well as their triglycerides and four sesquiterpene lac-

\*Part 451 in the series "Naturally Occurring Terpene Derivatives". For Part 450 see Salam, N. A. A., Mahmoud, Z. F., Ziesche, J. and Bohlmann, F. (1982) *Phytochemistry* **21**, 2746.