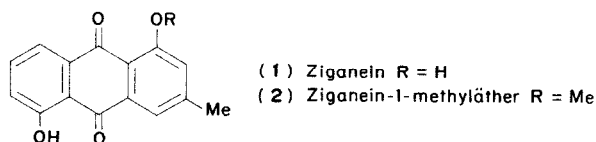


Bei Literaturdurchsuchung der Schmp.-Daten von α -Dihydroxy- β -methylantrachinone stellte man fest, dass das von Mühlemann² synthetisierte 1,5-Dihydroxy-3-methylantrachinon einen ähnlichen Schmp. wie **1** hat (222–223°).^{*} Wir verglichen dann beide Verbindungen durch Mischschmp., Cochromatographie, UV- und IR-Spektroskopie und es stellte sich heraus, dass sie vollkommen identisch sind. Somit wurde die Struktur von **1** festgelegt.

Von den beiden Monomethyläthern des 1,5-Dihydroxy-3-methylantrachinons ist nur der 5-Methyläther (Schmp. 179–180°) bekannt.² Der Vergleich von **2** mit dieser Verbindung (DC, UV- und IR-Spektroskopie) ergab keine Identität. Demnach kommt **2** die Konstitution des 5-Hydroxy-1-methoxy-3-methylantrachinons zu.



Für **1** und **2**, zwei erstmals aus der Natur isolierten Anthrachinone, wollen wir die Bezeichnungen Ziganein und Ziganein-1-methyläther vorschlagen.[†]

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^{*} Die Testsubst. schmolz entgegen der Lit. Angabe scharf bei 227°.

[†] Nach dem Fundort der Pflanze: Zigana Berg in Nordost Anatolien, Türkei.

² MÜHELMANN, H. (1949) *Pharm. Acta Helv.* **24**, 356.

Phytochemistry, 1974, Vol. 13, pp. 682 to 683. Pergamon Press. Printed in England.

WAXY β -DIKETONES FROM *SECALE CEREALE*

PAUL DIERICKX

Laboratory of Plant Physiology, Vaartstraat 24, Leuven, Belgium
and

FRANS COMPERNOLLE

Laboratory of Macromolecular Chemistry, Celestijnenlaan 200 F, Heverlee, Belgium

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Key Word Index — *Secale cereale*; Gramineae; rye; long chain β -diketones.

Plant. Secale cereale L. *Source*. The seeds were obtained from the Belgische Boerenbond, Leuven, Belgium. *Previous work*. 25-OH-*n*-C₃₁-14,16-dione was cited previously from *Triticum durum*.¹

¹ TULLOCH, A. P. and HOFFMAN, L. L. (1971) *Phytochemistry* **10**, 871.

Present work. Wax was extracted from whole fresh plants (920 g) and further analyzed as described earlier.² The β -diketone was identified as *n*-C₃₁-14,16-dione (TLC on silica gel G, C₆H₆, *R_f* 0.86; reaction with Fast Blue B salt; UV; IR; MS comparison with authentic sample).

The hydroxy- β -diketone is much more polar (*R_f* 0.08). It shows the same yellow reaction as the unhydroxylated compound with Fast Blue B salt and the same UV absorption maximum at 274 nm in Et₂O, but has additional IR-bands at 3340 and 1025 cm⁻¹, resulting from the secondary alcohol group. MS showed a molecular ion mass peak of 480 *m/e*, in agreement with the molecular formula C₃₁H₆₀O₃. Major ions were observed at 192, 211,³ 218, 235, 236, 237, 250, 253,³ 255, 268, 276, 279, 281, 294, 297, 312, 348, 366, 395, 409, 444 and 462 *m/e*; the peaks at 395 and 409 *m/e*, thus determine the position of the OH at C₂₅. No trace of isomers could be detected. This confirms the earlier statement that the OH is introduced on the C₁₅-side of the diketo moiety precisely at C₂₅,¹ whereas its introduction is not positionally specific in the C₁₃-part of the molecule.^{2,4}

As β -diketones have been found in the non-cereal grass *Festuca glauca*,⁵ we also examined the following grasses: *Lolium perenne* L., *Molinia caerulea* (L.) Moench, *Dactylis glomerata* L., *Phragmites communis* Trin., *Arrhenatherum elatius* P.B. ex J. et C., *Holcus mollis* L., *Corynephorus canescens* (L.) P.B., *Ammophila arenaria* (L.) Link. Although the spray reagent is very sensitive to compounds having a dicarbonyl group,² no trace of a β -diketone could be detected in the waxes of these grasses; we concluded, therefore, that β -diketones cannot be considered as chemotaxonomic markers in the Graminae.

² DIERICKX, P. J. and BUFFEL, K. (1972) *Phytochemistry* **11**, 2654.

³ JACKSON, L. L. (1971) *Phytochemistry* **10**, 487.

⁴ TULLOCH, A. P. and WEENINK, R. O. (1969) *Can. J. Chem.* **47**, 3119.

⁵ HORN, D. H. S. and LAMBERTON, J. A. (1962) *Chem. Ind. (London)* 2036.