

from ethyl acetate-butanol (1:1). The product, m.p. 165–175° (decomp.), showed $[\alpha]_D^{25} +75$ (c 1.0 water), with no mutarotation, indicating that it is a mixture of anomers, (Found: C 48.6; H 6.7; N 5.6. $C_{10}H_{17}NO_6$ requires C 48.6; H 6.9; N 5.7.)

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1. Holmquist, L. *Acta Chem. Scand.* **25** (1971) 712.
2. Pravdić, N. and Fletcher, Jr., H. G. *Carbohydr. Res.* **12** (1970) 471.
3. Wolfrom, M. L. and Wood, H. B. *J. Am. Chem. Soc.* **73** (1951) 2933.
4. Partch, R. E. *Tetrahedron Letters* **1964** 3071.
5. Kuhn, R. and Baschang, G. *Ann.* **659** (1962) 156.
6. Holmquist, L. *Acta Chem. Scand.* **23** (1969) 1045.

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On the Crystal Structure of $RhMg_{\sim 6}$

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According to Ferro the phases $PdMg_{5.6-6.9}$ ¹ and $PtMg_{\sim 6}$ ² are isotypic with a cell edge $a \approx 20.1$ Å. No further data on the crystal structure have been reported. In the course of phase analysis and crystal structure studies on the platinum metal-magnesium systems a similar compound has also been found in the Rh–Mg system. Phase analysis of powder patterns obtained from samples of different compositions

around $RhMg_6$ and examinations of single crystals showed that a crystal obtained from a sample somewhat higher in magnesium than 1:6 ratio ($RhMg_{6.3}$) was best suited for a further single crystal study.

The alloy $RhMg_{6.3}$ was prepared from weighed amounts of the components by melting in a sealed iron tube. The tube was heated to 1150°C, slowly cooled (3°C per hour) to 500°C and then quenched in water to room temperature. The specimen thus prepared showed no contamination of iron when chemically analyzed. The Guinier powder photograph of the product showed no extra lines and the pattern could be indexed assuming a cubic unit cell with $a = 20.148 \pm 1$ Å.

A single crystal formed as a cube (the edge less than 0.1 mm) was mounted on a single-crystal X-ray diffractometer and 710 independent reflections ($\theta \leq 31^\circ$) were collected with $MoK\alpha$ radiation. The systematic absences were consistent with space groups $Fm\bar{3}m$, $F\bar{4}3m$, and $F432$.

A trial structure was derived by using a Samson packing map³ obtained from the symmetry of the (110)-plane of space group $F\bar{4}3m$. The structure was assumed to be built up by icosahedra of magnesium atoms surrounding a rhodium atom. Regular icosahedra with an edge of 3 Å were fitted together on the packing map. In this way the approximate positions of three rhodium and eleven magnesium atoms were found.

The atomic parameters thus obtained were taken as starting values in a least squares refinement. An R -value of 0.085 was obtained. The final structural data corresponding to the formula Rh_7Mg_{44} are given in Table 1.

All the rhodium atoms are surrounded by twelve magnesium atoms forming an icosahedron. The Rh(1)–Mg, Rh(2)–Mg, and Rh(3)–Mg distances are in the ranges 2.65–3.16, 2.72–3.10, and 2.82–2.99 Å, respectively. Four icosahedra of Rh(3) share corners and these corners form an octahedron with the center at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$. This part of the structure is also found in the structure of $VALi_6$.⁴ Four icosahedra of Rh(2) share faces and form a complex of atoms similar to an arrangement found in the gamma phase structure.⁵ Six icosahedra of Rh(1) share faces and this arrangement results in very deformed icosahedra with a great variation in the Rh–Mg distances. The very short distance 2.65 Å may be compared with another short palladium

Table 1. Atomic coordinates and thermal parameters of Rh₇Mg₄₄.

Atom	Special positions	x	z	B (Å ²)
Rh(1)	in 24 g	1/4	0.5867 ± 1	1.03 ± 4
Rh(2)	in 16 e	0.0833 ± 1		0.70 ± 4
Rh(3)	in 16 e	0.3488 ± 1		0.68 ± 4
Mg(1)	in 48 h	0.0525 ± 3	0.3380 ± 4	1.1 ± 1
Mg(2)	in 48 h	0.1072 ± 3	0.2141 ± 4	1.0 ± 1
Mg(3)	in 48 h	0.1949 ± 3	0.4806 ± 4	0.7 ± 2
Mg(4)	in 48 h	0.0961 ± 3	0.7206 ± 5	1.4 ± 1
Mg(5)	in 48 h	0.1565 ± 3	0.9795 ± 4	1.0 ± 1
Mg(6)	in 24 g	1/4	0.3553 ± 6	0.9 ± 1
Mg(7)	in 24 f	0	0.1824 ± 7	1.2 ± 2
Mg(8)	in 16 e	0.6956 ± 4		0.7 ± 2
Mg(9)	in 16 e	0.8338 ± 4		0.5 ± 2
Mg(10)	in 16 e	0.5618 ± 6		1.8 ± 3
Mg(11)	in 16 e	0.9476 ± 4		0.9 ± 2

metal-magnesium contact found in Pd₂Mg₅, viz. 2.59 Å.⁶ A detailed description of this structure will be published subsequently.

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1. Ferro, R. *J. Less Common Metals* **1** (1959) 424.
2. Ferro, R. *J. Less Common Metals* **2** (1960) 383.
3. Samson, S. *Acta Cryst.* **17** (1964) 591.
4. Brown, P. J. *Acta Cryst.* **10** (1957) 133.
5. Westman, S. *Acta Chem. Scand.* **19** (1965) 1411.
6. Westin, L. *Acta Chem. Scand.* **22** (1968) 2574.

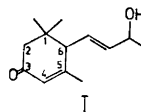
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Tobacco Chemistry 7. Structure and Synthesis of 3-Oxo- α -ionol, a New Tobacco Constituent

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During the chemical study of the volatile constituents of sun-cured Greek tobacco, *N. tabacum* grown in Serres 1968, we have isolated a new compound (14 ppm of dry tobacco) which has been assigned structure I on the basis of evidence presented below and is now named 3-oxo- α -ionol.



Accurate mass measurements suggested that I had the composition C₁₃H₂₀O₂. (Found 208.1457, calc. 208.1463.) The UV spectrum in ethanol exhibited a maximum at 235 nm (ϵ 9000) indicating the presence of a disubstituted conjugated ketone grouping. This assignment was supported by a band at 1657 cm⁻¹ in the IR spectrum (film) which also displayed diagnostically important bands at 3400 cm⁻¹ (hydroxyl), 1369 cm⁻¹ and 1376 cm⁻¹ (*gem.* dimethyl) and 976 cm⁻¹ (*trans* disubstituted double bond). The NMR results (60 MHz, CDCl₃, TMS) are summarised in Table 1.

Decoupling experiments revealed that the protons resonating at 1.91 and 5.96 ppm are coupled (J 1.4 Hz), consistent with the presence of a -CO-CH=C(CH₃)- group. The protons giving rise to the signals at ca. 4.4 and 1.31 ppm were similarly shown to be coupled (J 6.3 Hz) to each other and can readily be associated with

a $\begin{array}{c} | \\ -C=C- \\ | \end{array}$ -CH(OH)-CH₃ moiety. The broad signal at 2.4 ppm disappeared on addition of D₂O and was thus due to a hydroxyl proton. The broad singlets at 2.17 ppm (*ca.* 1H) and 2.26 ppm (*ca.* 1H) seem to form the inner lines of an AB-

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