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Publisher *Taylor & Francis*

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Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

ESTERS OF (\pm)HEXAHYDRO-5-HYDROXYMETHYL-2H-AZEPIN-2-ONE

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To cite this Article Overberger, C. G. and Kozolwski, J. H. (1973) 'ESTERS OF (\pm)HEXAHYDRO-5-HYDROXYMETHYL-2H-AZEPIN-2-ONE', *Organic Preparations and Procedures International*, 5: 4, 199 – 202

To link to this Article: DOI: 10.1080/00304947309355568

URL: <http://dx.doi.org/10.1080/00304947309355568>

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picrate salt, best prepared by treatment with excess saturated methanolic picric acid followed by decomposition of the pure picrate, mp. 123^o, with aqueous base.

REFERENCES

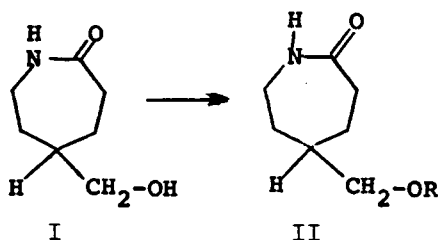
- 1.- M. Davis and A. W. White, Chem. Comm., 1547 (1968).
- 2.- M. Davis and A. W. White, J. Org. Chem., 34, 2985 (1969).

ESTERS OF (±)HEXAHYDRO-5-HYDROXYMETHYL-2H-AZEPIN-2-ONE

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(6/7/73)

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Five new derivatives (II) of (±)hexahydro-5-hydroxymethyl-2H-azepin-2-one (I)¹ have been prepared and characterized.



II, R =

- a) 1-Menthoxyacetyl
- b) Phthaloyl-1-phenylalanyl
- c) D-10-Camphorsulfonyl
- d) Phthaloyl-H
- e) Succinoyl-H

EXPERIMENTAL⁵

(±)Hexahydro-5-(1-menthoxyacetoxymethyl)-2H-azepin-2-one (IIa).

A solution of 6.6 g (0.028 mole) 1-menthoxyacetyl chloride² [α]_D²⁴ -83.7^o (c, 2.0 chloroform), (bp. 67^o/0.04 mm),

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in 35 ml of ethanol-free chloroform was added dropwise to a stirred solution of I, 4 g (0.028 mole) in 45 ml of pyridine at 0°. The mixture was stirred for 3 hr at 0° and then for 9-10 hrs at room temperature. The pyridine and chloroform were removed under reduced pressure. The residue was dissolved in chloroform and washed with 1 N HCl, then 1 N sodium bicarbonate and finally with water. Evaporation of the chloroform yielded a residue which was crystallized from *n*-pentane to give 8.7 g (90%) of a white solid, mp. 54-59°; the ester is soluble in most organic solvents.

(±)Hexahydro-5-(phthaloyl-*l*-phenylalanyloxymethyl)-2H-azepin-2-one (IIb). - The compound was prepared as described for IIa from 3.14 g (0.01 mole) of phthaloyl-*l*-alanyl chloride³ (mp. 82-83°, $[\alpha]_D^{25}$ -96° (c, 2.2 benzene)), and 1.43 g (0.01 mole) of I. Removal of the chloroform at the last step gave an analytical sample as a colorless solid, mp. 152-158°.

(±)Hexahydro-5-(D-10-camphorsulfonyloxymethyl)-2H-azepin-2-one (IIc). - The compound was prepared as described for IIa from 10 g of D-10-camphorsulfonyl chloride⁴ (mp. 67-68°) and 5 g of I except for the work up which consisted of pouring the reaction mixture into 40 ml of water, 100 g of ice and 5.6 g of conc. HCl. Recrystallization of the white powder from *n*-propanol gave needles, mp. 143.5-144.5°.

(±)Hexahydro-5-(hydrogen phthaloyloxymethyl)-2H-azepin-2-one (IIId). - Phthalic anhydride, 14.8 g (0.1 mole), and (±)hexahydro-5-hydroxymethyl-2H-azepin-2-one, 14.3 g (0.1 mole), were dissolved in 125 ml of pyridine on a steam bath and heated for

12 hrs. The pyridine was removed under reduced pressure leaving a white solid. The solid was dissolved in a solution of sodium carbonate and extracted twice with 100 ml portions of chloroform. The addition of excess 4 N HCl precipitated the solid. Recrystallization from 1100 ml of n-propanol gave 25 g (86%) of fine white needles, mp. 198-201° (dec.).

(±)Hexahydro-5-(hydrogen succinoyloxymethyl)-2H-azepin-2-one (IIe). - The compound was prepared as described for IIId from 3 g of succinic anhydride and 4.3 g of I. However, the viscous liquid obtained after evaporation could not be induced to crystallize although it gave a correct elemental analysis. IR(neat): 3280, 3150-2500, 2930, 1640, 1635, 1170 cm^{-1} .

Elemental Analyses

	<u>Calculated</u>			<u>Found</u>		
	C,	H,	N	C,	H,	N
IIa	67.22	9.80	4.13	67.27	9.74	4.17
IIb	61.85	5.88	4.81	61.84	5.89	4.81
IIc ^a	57.13	7.61	3.92	51.31	7.35	3.92
IIId	68.56	5.75	6.66	68.44	5.86	6.49
IIe	54.31	7.04	5.76	54.11	7.30	5.62

a) Calcd.: S, 8.95. Found: S, 9.00.

Acknowledgement. - We gratefully acknowledge support of this work by the Chemistry Department of The University of Michigan, the Dow Chemical Company, the National Science Foundation traineeship, Hercules Incorporated, Sherwin-Williams-VanStone fellowship, and the Union Carbide Corporation. We also wish to thank Dr. James A. Moore for his help in identifying these preparations.

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REFERENCES

1. C. G. Overberger and J. H. Kozlowski, J. Polym. Sci., A-1, 10, 2265, 2291 (1972).
2. D. F. Holmes and R. Adams, J. Amer. Chem. Soc., 56, 2093 (1934).
3. J. C. Sheehan, D. W. Chapman, and R. W. Roth, J. Amer. Chem. Soc., 74, 3822 (1952).
4. S. Smiles and T. P. Hilditch, J. Chem. Soc., 91, 522 (1907).
5. Bps. and mps. are uncorrected. Microanalyses were performed by Alfred Bernhardt Microanalytisches Laboratorium, Mulheim (Ruhr), West Germany and by Spang Microanalytical Laboratory, Ann Arbor, Michigan. A Bendix-Ericsson Type 143A polarimeter was used in determining optical activity at the sodium D line.