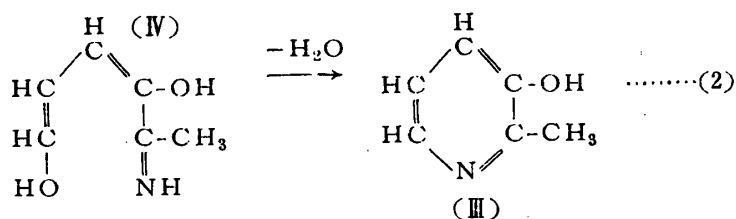


FORMATION OF 2-METHYL-3-HYDROXY-PYRIDINE FROM FURYL-METHYLKETONE

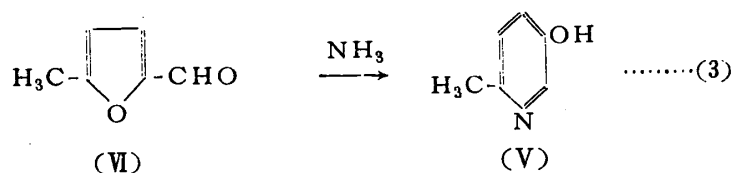
著者	ASO Kiyoshi
journal or publication title	Tohoku journal of agricultural research
volume	1
number	1
page range	125-127
year	1950-03
URL	http://hdl.handle.net/10097/29028



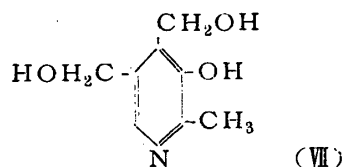
The crystal obtained is a colorless prism, melts at 167-168°, and indicates a deep red color by FeCl₃, and its picrate melts at 202-203° (decomposes).

Because its properties are similar to 2-methyl-5-hydroxy-pyridine (V) obtained⁽²⁾ from 5-methyl-furfural (VI) and NH₃-salt by same reaction (3), the author measured the melting points of these mixed crystals.

The resulting melting points were dropped 11° and 12° (picrate) respectively. The two crystals, therefore, are not same material.



But Dornow⁽³⁾ synthesizes 2-methyl-3-hydroxy-pyridine (III) from a standpoint of similar constitution of vitamin B₆ (VII), and reports that it has not a vitamin B₆-like activity.



It is reported that the crystal (III) is a colorless prism, melts at 167-8° and indicates deep red color by FeCl₃, and its picrate melts at 204° (decomposes).

Though (III) was not synthesized, the author believes the crystals obtained from fural-methylketone to be (III) from the fact presented above.

Experimental part

Fural-methylketone 4g, ammonium sulfate 5g and H₂O 50cc are heated in an autoclave at a temperature of 160-161° for 2 hours. After cooling, the solution is filtered, concentrated to half of original volume under reduced pressure, alkalified with sodium carbonate, and extracted with ether.

Yellow crystals are obtained by removing ether from the extract, which is then recrystallized from benzol after one sublimation.

It is a colorless prism, melts at 167-8°, and indicates a deep red color by FeCl₃.

Microanalysis.

Sample (mg)	N (cc)	b (mm)	t° (c)	N (%)
2.738	0.311	758	15	13.16
2.615	0.301	759	16	13.31
C ₆ H ₇ ON theoretically				12.84

Its picrate is obtained by addition of picric acid ether solution and is a yellow needle and melts at 202°-3° (decomposes),

Microanalysis.

Sample (mg)	N (cc)	b (mm)	t° (c)	N (%)
2.764	0.401	758	15	16.81
2.674	0.384	759	16	16.60
C ₆ H ₇ ON. C ₆ H ₂ (OH)(NO ₂) ₃ ... theoretically ...				16.57

Acknowledgment.

I express my deep gratitude to Dr. Teijiro Yabuta for his kind guidance and I wish to thank Mr. Teiichi Tamura for his synthesized furyl-methylektone.

Literature

- (1) The Bulletin of the Institute of Physical and Chemical Research, Vol. 18, 171-176 (1939). (Japanese report).
- (2) Ibid. report, Vol. 18, 182-184 (1939).
- (3) Dornow. 1940. Ber., 73, 78-80.