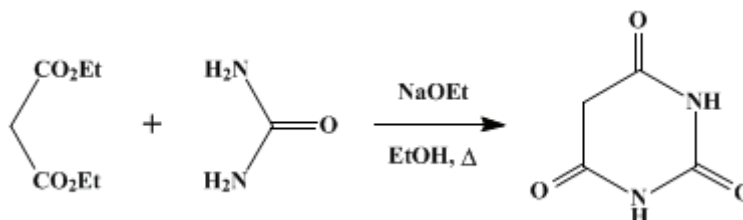


## BARBITURIC ACID



Submitted by J. B. Dickey and A. R. Gray.  
Checked by Reynold C. Fuson and W. E. Ross.

### 1. Procedure

In a 2-l. round-bottomed flask fitted with a reflux condenser protected by a calcium chloride tube, 11.5 g. (0.5 gram atom) of finely cut sodium is dissolved in 250 cc. of absolute alcohol. To this solution is added 80 g. (0.5 mole) of ethyl malonate followed by 30 g. (0.5 mole) of dry urea dissolved in 250 cc. of hot (70°) absolute alcohol. After being well shaken the mixture is refluxed for seven hours on an oil bath heated to 110°. A white solid separates rapidly. After the reaction is completed, 500 cc. of hot (50°) water is added and then enough hydrochloric acid (sp. gr. 1.18) to make the solution acidic (about 45 cc.). The resulting clear solution is filtered and cooled in an ice bath overnight. The white product is collected on a Büchner funnel, washed with 50 cc. of cold water, and then dried in an oven at 105–110° for three to four hours. The yield of barbituric acid is 46–50 g. (72–78 per cent of the theoretical amount).

### 3. Discussion

Barbituric acid has been prepared by the action of phosphorus oxychloride on malonic acid and urea;<sup>1</sup> by treating an acetic acid solution of urea and malonic acid with acetic anhydride;<sup>2</sup> from ethyl malonate and urea using sodium ethoxide as a condensing agent;<sup>3</sup> and from ethyl malonate and the sodium derivative of urea prepared from urea and sodium in liquid ammonia.<sup>4</sup>

The procedure described is an adaption of that of Michael.<sup>3</sup>

This preparation is referenced from:

- *Org. Syn. Coll. Vol. 2*, 440
- *Org. Syn. Coll. Vol. 3*, 37

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### References and Notes

1. Grimaux, *Compt. rend.* **87**, 752 (1878); Conrad and Guthzeit, *Ber.* **14**, 1643 (1881); Grimaux, *Bull. soc. chim.* (2) **31**, 146 (1879); Matignon, *Ann. chim. phys.* (6) **28**, 289 (1893).
  2. Biltz and Wittek, *Ber.* **54**, 1035 (1921).
  3. Michael, *J. prakt. Chem.* (2) **35**, 456 (1887); Tafel and Weinschenk, *Ber.* **33**, 3383 (1900); Gabriel and Colman, *ibid.* **37**, 3657 (1904).
  4. Jacobson, U. S. pat. 2,090,594 [C. A. **31**, 7068 (1937)].
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**(Registry Number)**

sodium derivative of urea

alcohol (64-17-5)

hydrochloric acid (7647-01-0)

acetic acid (64-19-7)

ammonia (7664-41-7)

acetic anhydride (108-24-7)

Phosphorus Oxychloride (21295-50-1)

sodium (13966-32-0)

sodium ethoxide (141-52-6)

urea (57-13-6)

ethyl malonate (1071-46-1)

Malonic acid (141-82-2)

Barbituric acid (67-52-7)