54% yield, and melted at 153.5–155.0° after recrystallization from acetone.

Anal. Calcd. for $C_{11}H_{21}NO_2 \cdot HCl$: C, 56.0; H, 9.4; N, 5.9. Found: C, 56.1; H, 9.3; N, 5.7.

(1-Acetoxymethylcyclohexyl)-trimethylammonium Iodide (III).—The methiodide was prepared from the hydrochloride as described in method B above; m.p. 169.0–170.0° (recrystallized from isopropyl alcohol), 81% yield.

Anal. Calcd. for $C_{12}H_{24}INO_2$: C, 42.2; H, 9.1. Found: C, 42.2; H, 9.0.

[β-(N,N-Diethylthiocarbamyloxy)-*t*-butyl]-trimethylammonium Iodide.—Reaction of II and N,N-diethylthiocarbamyl chloride according to method J yielded 2-dimethylamino-2-methylpropyl N,N-diethylthiocarbamate, b.p. 114–118° at 3 mm., $n^{25}D$ 1.5002.

Anal. Calcd. for $C_{11}H_{24}N_2OS$: C, 56.8; H, 10.4; N, 12.1. Found: C, 57.5; H, 10.6; N, 11.4.

The methiodide melted at 187.5–189.0° after recrystallization from isopropyl alcohol–ethyl acetate.

Anal. Calcd. for $C_{12}H_{27}IN_2OS$: C, 38.5; H, 7.3. Found: C, 39.4; H, 7.6.

SYRACUSE, NEW YORK

NOTES

8-Basically-substituted Caffeines

BY F. F. BLICKE AND H. C. GODT, JR.

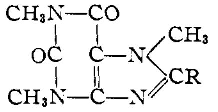
RECEIVED JANUARY 11, 1954

During a study of substituted xanthines, a few 8-basically substituted caffeines[1] were prepared by amination of 8-chlorocaffeine.

Experimental

General Procedure.—8-Chlorocaffeine[2] (11.4 g., 0.05 mole), 0.1 mole of the required amine and 75 cc. of absolute ethanol were heated in a citrate bottle at 150° for 6 hours. After refrigeration for 12 hours, the precipitate was filtered and recrystallized.

8-BASICALLY-SUBSTITUTED CAFFEINES



All compounds were recrystallized from absolute ethanol except 3 which was recrystallized from 50% methanol.

	R	M.p., °C.	Yield, %	Formula	Carbon, % Calcd.	Carbon, % Found	Hydrogen, % Calcd.	Hydrogen, % Found	Nitrogen, % Calcd.	Nitrogen, % Found
1	Pyrrolidino	184–186	76	$C_{12}H_{17}O_2N_5$	54.74	54.54	6.51	6.51	26.60	26.48
2	Piperidino	141–143[a]	75	....	...	...	..	..	...	...
3	1-Hexamethylenimino	114–116	69	$C_{14}H_{21}O_2N_5$	57.71	57.97	7.27	7.42	24.04	24.32
4	Morpholino	166–167	66	$C_{12}H_{17}O_3N_5$	51.60	51.41	6.14	6.15	25.08	25.28
5	β-Phenylethylamino	219–221	74	$C_{16}H_{19}O_2N_5$	61.33	61.59	6.11	6.39	22.35	22.39

[a] Ref. 1c, m.p. 142°.

Three of the products listed in the table were tested for diuretic activity in the Lilly Research Laboratories. Compound 1, administered orally, produced only slight diuresis in two of six dogs (200 mg. dose). Tested in the same manner, compound 3 (400 mg. dose) and compound 4 (100 mg. dose) did not produce diuresis.

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[1] Other 8 basically-substituted caffeines, in which the basic nitrogen atom is attached directly to the 8-carbon atom, have been described by (a) E. Fischer (*Ann.*, **215**, 253 (1882)), (b) L. Cramer (*Ber.*, **27**, 3098 (1894)), (c) A. Einhorn and E. Baumeister (*ibid.*, **31**, 1138 (1898)) and (d) M. Gomberg (*Am. Chem. J.*, **23**, 51 (1900)).

[2] L. M. Long, THIS JOURNAL, **69**, 2939 (1947).

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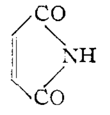
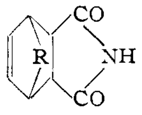
A Synthesis of Maleimide

BY JEROME A. BERSON AND RONALD SWIDLER

RECEIVED DECEMBER 30, 1953



I



II

Maleimide (I), a relatively simple substance, has proved to be remarkably inaccessible. While various N- and C-substituted derivatives of I have been prepared by straightforward methods,[1] only two practical procedures for the preparation of I itself have been reported. Plancher and Cattadori[2] obtained I in small yield by chromic acid oxidation of pyrrole. Prill[3] employed a dienophile exchange reaction in which bicyclic imide adducts of the type II were heated in a gas flow system with excess maleic anhydride, while Tawney[4] effected similar decompositions without the use of an added diene acceptor.

The present synthesis of I takes advantage of the particularly facile retrogression of the Diels–Alder reaction in the furan series and of our recently described[5] procedure for the preparation of II (R = O) from the readily accessible furan–maleic anhydride adduct (III).

The steps of the sequence III → IV → V → II (R = O) proceed in yields of 91, 89 and 88%, respectively. Upon being heated at 180–190°, II

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[1] (a) A. Piutti and E. Giustiniani, *Gazz. chim. ital.*, **26**, I, 435 (1896); (b) J. Gottlieb, *Ann.*, **77**, 274 (1851); (c) G. Ciamician and M. Dennstedt, *Gazz. chim. ital.*, **12**, 501 (1882).

[2] (a) G. Plancher and V. Cattadori, *Atti della Reale Acad. dei Lincei*, **13**, I, 490 (1904); (b) H. Kwart and I. Burchuk, THIS JOURNAL, **74**, 3094 (1952).

[3] E. J. Prill, U. S. Patent 2,524,136 (1950).

[4] P. O. Tawney, U. S. Patent 2,524,145 (1950). We are indebted to a referee for pointing out this reference.

[5] J. A. Berson and R. Swidler, THIS JOURNAL, **76**, in press.