TABLE I

MORPHOLINOMETHYL KETONES

Ketone, M = morpholino	B. p., °C. mm.	M. p., °C.	Yield %	Nitrogen, % Calcd.	Nitrogen, % Found
M-acetone	101–101.5 (14)	....	37	9.79	9.74
1-M-butanone-2	97–100 (9)	....	38	8.92	8.74
ω-M-acetophenone	.....	50–52	..	6.83	6.27
ω-M-*p*-phenylacetophenone	.....	113–114	95	4.98	4.80
ω-M-*p*-bromoacetophenone	.....	88.5–89	52	4.93	4.61

TABLE II

HYDROCHLORIDES AND PICRATES OF MORPHOLINOMETHYL KETONES

Ketone, M = morpholino	Hydrochlorides M. p., °C.	Hydrochlorides Chlorine, % Calcd.	Hydrochlorides Chlorine, % Found	Picrates M. p., °C.	Picrates Nitrogen, % Calcd.	Picrates Nitrogen, % Found
M-acetone	183	19.75	19.91	145.5	15.06	15.05
1-M-butanone-2	171–172.5	18.32	18.29	127–129	14.51	13.89
ω-M-acetophenone	212–214[a]	...	...	156–157	12.91	12.89
ω-M-*p*-phenylacetophenone	233–235[b]	11.16	10.50	160–162	10.98	10.73
ω-M-*p*-bromoacetophenone	218 (dec.)	11.06	11.18	145–146	10.92	10.94

[a] Rubin and Day[2] obtained a melting point of 222–223°. [b] ω-Morpholino-*p*-phenylacetophenone hydrobromide melted at 233–234°. *Anal.* Calcd. for $C_{18}H_{20}BrNO_2$: N, 3.87. Found: N, 3.64.

ume of anhydrous ether approximately ten times the volume of the chloro ketone. The mixture was allowed to stand for twenty-four hours, or preferably shaken mechanically for this period. The solid morpholine hydrochloride was removed by filtration and the ether removed by distillation. During this process a small additional precipitate of morpholine hydrochloride appeared and was filtered off. The residue was distilled under reduced pressure and the results are given in Table I. After redistillation under reduced pressure, the products were colorless liquids which gradually became yellow on standing for several weeks. Samples which distilled over a range of more than two degrees changed to red in color in a few days.

ω-Morpholinoacetophenone was obtained from the hydrochloride[2] by treating the aqueous solution of the hydrochloride with solid sodium hydroxide, filtering, and washing the precipitate well with water. The white solid was dried in a vacuum desiccator. It was analyzed as quickly as possible after drying, because the solid gradually became yellow, and after standing four or five days it was a dark viscous oil.

ω-Morpholino-*p*-phenylacetophenone and ω-morpholino-*p*-bromoacetophenone were made by the method used by Rubin and Day.[2] In each case, however, it was found that the solid which precipitated after the reaction mixture had stood overnight was a mixture of morpholine hydrochloride and the corresponding morpholino ketone. The mixture was suspended in water to dissolve the morpholine hydrochloride. The morpholino ketone was removed by filtration, washed with water and recrystallized from alcohol. The results are given in Table I.

The hydrochlorides were made by passing dry hydrogen chloride into ether or benzene solutions of the ketones. The hydrochlorides of morpholinoacetone, 1-morpholinobutanone-2 and ω-morpholinoacetophenone were recrystallized from alcohol solution by the addition of ether. The hydrochlorides of ω-morpholino-*p*-phenylacetophenone and ω-morpholino-*p*-bromoacetophenone were recrystallized from alcohol (see Table II).

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[2] Rubin and Day, *J. Org. Chem.*, **5**, 54 (1940).

p-BROMOPHENYLCYCLOPENTANE

Twenty grams of bromine was added to 20 g. of phenylcyclopentane and 2 g. of iodine as in the method described by Bodroux and Thomassin[1] for the bromination of phenylcyclohexane. The addition took thirty minutes and the mixture was then allowed to stand another thirty minutes. Vigorous evolution of hydrogen bromide was observed during the entire process. The solution was then treated with sodium bisulfite solution to get rid of excess halogens. Benzene was then added to dissolve the oil and the solution was dried over calcium chloride. The benzene was distilled off and the residual oil vacuum distilled. About thirteen grams of unchanged phenylcyclopentane was recovered. Six grams of the desired product was obtained boiling at 115–118° at twenty millimeters pressure, yield 55%. The substance when freshly distilled is a colorless, fragrant oil, but on standing acquires a light brown color; d^{20}_{20} 1.3175, $n^{20}\text{D}$ 1.5642, M_D calcd. 55.26, M_D observed 55.72.

Anal. Calcd. for $C_{11}H_{13}Br$: Br, 35.5. Found: Br, 35.56.

The structure of this compound was established by oxidation with sodium dichromate. The acid isolated, melting point 250°, was *p*-bromobenzoic acid.

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[1] Bodroux and Thomassin, *Bull. soc. chim.*, [5] **6**, 1411 (1939).