

and sucked dry. The filter cake was removed, triturated with 50 ml acetone, and filtered. Yield and other data are given in Table I.

For the preparation of compounds **IVa** and **b**, respectively, pyruvic aldehyde (methylglyoxal) was used as the 40% aqueous solution and phenylglyoxal as the monohydrate. These aldehydes required a reaction time of 30–45 min.

The ir spectra of all compounds agreed with the assigned structures, with secondary amide absorption being noted in the ranges 1530–1580, 1630–1660, and 3280 cm^{-1} . In addition, compounds **IIIe–i** and **IVa** and **b** gave bands characteristic of the respective aromatic substitution. Compound **IVa** showed absorption at 1725 and 1370 cm^{-1} , characteristic of the carbonyl and acetyl groups, respectively. **IVb** gave carbonyl absorption at 1690 cm^{-1} .

Confirmatory proton nmr spectra were obtained as follows: **IVa**: (DMSO- d_6), δ 2.25 (s, CH_3CO), 6.17–5.92 (t, —CH, $J = 7$ Hz), 8.03–7.40 (m, 10H, C_6H_5), 9.17–9.03 (d, 2H, —NH, $J = 7$ Hz). **IVb**: (DMSO- d_6), δ 7.15–7.04

(t, —CH, $J = 7$ Hz), 8.12–7.42 (m, 15H, C_6H_5), 9.30–9.18 (d, 2H, —NH, $J = 7$ Hz).

Attempts to obtain mass spectral data were unsuccessful because of the instability of the compounds at the required temperature.

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Literature Cited

- (1) Drach, B. S., Dolgushina, I. Yu., Kirsanov, A. V., *Zh. Org. Khim.* **8** (6), 1224 (1972); *CA*, **77**, 101055 (1972).
- (2) Gilbert, E. E., *Synthesis*, **1972**, p 30.
- (3) Gilbert, E. E., *ibid.*, p 136.
- (4) Khasapov, B. N., Novikova, T. S., Lebedev, O. V., Khmel'nitskii, L. I., Novikov, S. S., *Zh. Org. Khim.* **8** (10), 2013 (1972); *CA*, **78**, 42989 (1973).
- (5) Nematollahi, J., Ketcham, R., *J. Org. Chem.* **28**, 2378 (1963).
- (6) Slezak, F. B., Bluestone, H., Magee, T. A., Wotiz, J. H., *ibid.* **27**, 2181 (1962).
- (7) Vail, S. L., Moran, C. M., Barker, R. H., *ibid.* **30**, 1195 (1965).

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Synthesis of *N*-Aryl-*N'*-2-naphthiazolyguanidines

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The experimental conditions for the synthesis of naphthiazolyguanidines are given.

Because guanidine derivatives possess a high degree of biological activity (1, 2), it was thought worthwhile to synthesize some naphthiazolyguanidines.

Experimental

***N*-Phenyl-*N'*-2-naphthiazolythiocarbamide.** Phenylisothiocyanate (3 ml) and 2-aminonaphthiazole (5 grams) were refluxed for 2 hr. The excess phenylisothiocyanate and 2-aminonaphthiazole were removed by washing several times with petroleum ether and ether. The thiocarbamide thus obtained was crystallized from EtOH, mp

161°, yield 75%. Similarly, various substituted *N*-aryl-*N'*-2-naphthiazolythiocarbamides were prepared (Table I).

***N*-Phenyl-*N'*-2-naphthiazolyguanidine.** *N*-phenyl-*N'*-2-naphthiazolythiocarbamide quantitatively formed the corresponding guanidine when heated with yellow lead oxide and ethanolic NH_3 in a sealed tube at 110°C. The product was crystallized from 50% EtOH, mp 185°. Similarly, various aryl-substituted *N*-aryl-*N'*-2-naphthiazolyguanidines and their hydrochlorides were prepared (Table II).

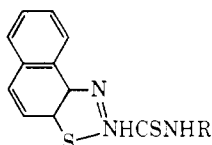
Literature Cited

- (1) Rose, F. L., Swain, G., *J. Chem. Soc.*, **1956**, p 4422.
- (2) Singh, G. C., *J. Indian Chem. Soc.*, **45**, 27 (1968).

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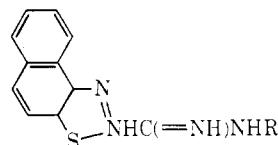
Table I. *N*-Aryl-*N'*-2-naphthiazolythiocarbamides



S No.	Nature of R	Mp, °C	Molecular formula ^a
1	<i>o</i> -MeC ₆ H ₄	135	C ₁₉ H ₁₅ N ₃ S ₂
2	<i>m</i> -MeC ₆ H ₄	132	C ₁₉ H ₁₅ N ₃ S ₂
3	<i>p</i> -MeC ₆ H ₄	158	C ₁₉ H ₁₅ N ₃ S ₂
4	<i>o</i> -OMeC ₆ H ₄	141	C ₁₉ H ₁₅ N ₃ OS ₂
5	<i>m</i> -OMeC ₆ H ₄	135	C ₁₉ H ₁₅ N ₃ OS ₂
6	<i>p</i> -OMeC ₆ H ₄	120	C ₁₉ H ₁₅ N ₃ OS ₂
7	<i>p</i> -OEtC ₆ H ₄	159	C ₂₀ H ₁₇ N ₃ OS ₂
8	<i>p</i> -ClC ₆ H ₄	164	C ₁₈ H ₁₂ ClN ₃ S ₂

^a All compounds analyzed satisfactorily for N,S.

Table II. *N*-Aryl-*N'*-2-naphthiazolyguanidines



S No.	Nature of R	Mp, °C	Molecular formula ^a	Mp, °C, hydrochloride	Molecular formula, hydrochloride
1	<i>o</i> -MeC ₆ H ₄	141	C ₁₉ H ₁₆ N ₄ S	253–54	C ₁₉ H ₁₇ ClN ₄ S
2	<i>m</i> -MeC ₆ H ₄	177	C ₁₉ H ₁₆ N ₄ S	185	C ₁₉ H ₁₇ ClN ₄ S
3	<i>p</i> -MeC ₆ H ₄	175	C ₁₉ H ₁₆ N ₄ S	204–205	C ₁₉ H ₁₇ ClN ₄ S
4	<i>o</i> -OMeC ₆ H ₄	175	C ₁₉ H ₁₆ N ₄ OS	195	C ₁₉ H ₁₇ ClN ₄ OS
5	<i>m</i> -OMeC ₆ H ₄	169	C ₁₉ H ₁₆ N ₄ OS	180	C ₁₉ H ₁₇ ClN ₄ OS
6	<i>p</i> -OMeC ₆ H ₄	171	C ₁₉ H ₁₆ N ₄ OS	190	C ₁₉ H ₁₇ ClN ₄ OS
7	<i>p</i> -OEtC ₆ H ₄	165	C ₂₀ H ₁₈ N ₄ OS	185	C ₂₀ H ₁₉ ClN ₄ OS
8	<i>p</i> -ClC ₆ H ₄	175	C ₁₈ H ₁₃ ClN ₄ S	181–82	C ₁₈ H ₁₄ Cl ₂ N ₄ S

^a All compounds analyzed satisfactorily for N,S.