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## Note

### Thin-layer chromatography of potential antifertility agents: N-aryl-N'-2-(benzoylbenzofuranyl)thiosemicarbazones

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Considerable interest recently has been focused on the synthesis and biological evaluation of substituted thiosemicarbazones that have been shown to possess antifertility effects. The thiosemicarbazones are of interest to the pharmacologist because of their wide spectrum of biological activity, and their unique physical and chemical properties make the compounds potential tool in drug receptor studies. A large number of substituted thiosemicarbazones<sup>1,2</sup> have been reported to possess powerful antispermatogenic activity. Prescott and Li<sup>3</sup> have shown that the thiosemicarbazones possess low toxicity. These observations prompted synthesis of some N-aryl-N'-2-(benzoylbenzofuranyl)thiosemicarbazones as potential antifertility agents, and a series these compounds were found to possess antispermatogenic activity. However, little or no information was available on their separation, identification and quantitation. Hence the present study was undertaken to establish a sensitive and reproducible chromatographic procedure for the separation and identification of various N-aryl-N'-2-(benzoylbenzofuranyl)thiosemicarbazones.

## MATERIALS AND METHODS

Table I illustrates the various N-aryl-N'-2-(benzoylbenzofuranyl)thiosemicarbazones analogues studied. They were synthesized by methods reported in literature<sup>4</sup>.

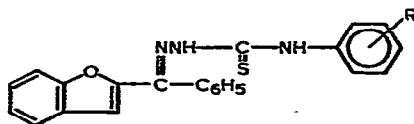
Thin-layer chromatographic (TLC) plates were prepared from a slurry of 40 g of silica gel G (Merck, Darmstadt, G.F.R.) in 80 ml of distilled water. The slurry was spread on 20 cm × 20 cm glass plates to a thickness of 0.20 mm with a Stahl's applicator. The plates were air-dried, activated at 110° for 3 h and stored in a desiccator.

A 1% methanolic solution of each compound was prepared and 1 μl of the solution (corresponding to 10 μg of each compound) was spotted at 2.0 cm from the

TABLE I

## THIN-LAYER CHROMATOGRAPHY OF N-ARYL-N'-2-(BENZOYLBENZOFURANYL)-THIOSEMICARBAZONES

General formula of compounds:



Detection:  $D_1$ , ferric chloride;  $D_2$ , basic lead acetate. Colour code: B, blue; Br, brown; D, dark; L, light; Or, orange; P, pink; R, red; V, violet; Y, yellow.

R	$R_f \times 100^*$	Detection	
		$D_1$	$D_2$
H	47	Y	DBr
2-CH <sub>3</sub>	43	Or	DBr
3-CH <sub>3</sub>	26	YOr	Br
4-CH <sub>3</sub>	23	OrR	DBr
2-OCH <sub>3</sub>	33	LY	DBr
3-OCH <sub>3</sub>	62	LV	DBr
4-OCH <sub>3</sub>	76	DP	DBr
2-Cl	50	DP	Br
3-Cl	36	Y	Br
4-Cl	20	P	DBr
2-OC <sub>2</sub> H <sub>5</sub>	48	B	DBr
4-OC <sub>2</sub> H <sub>5</sub>	29	LY	DBr
2,5-(CH <sub>3</sub> ) <sub>2</sub>	39	YOr	DBr

\* Average of four identical runs.

edge of the plate. The plate was developed at 32° using the solvent system benzene-chloroform-methanol (9:3:1) until the solvent front had travelled a distance of 16 cm. Usually, *ca.* 45 min were required for the development of the plate.

The plate was dried, sprayed with butanolic ferric chloride solution (1% w/v) and then heated at 50°. These compounds have also been detected with aqueous basic lead acetate (2% w/v) and heated at same temperature. Each of the compounds appears as shown in Table I.

## RESULTS AND DISCUSSIONS

The chromatographic results are shown in Table I. The detection limit was found to be *ca.* 1  $\mu$ g for each compound. The only adsorbent used was silica gel G. Activation of TLC plates at various temperatures (75–150°) was also examined, but no variation with temperature was observed. Several developing solvents, benzene, benzene-methanol (4:1), benzene-chloroform (6:4), benzene-ethanol (8:2), benzene-methanol-hexane (7:1:2) and chloroform, were examined.

Heating of the plates had little effect on the chromatograms, but the best separation was achieved with a plate heated at 110° for 30 min. On the other hand, the various developing solvents showed much greater differences, and sharp spots free from tailing were found only in the solvent system benzene-chloroform-methanol

(9:3:1). Increasing the proportion of methanol gave higher  $R_F$  values for these compounds but did not improve the separation.

The  $R_F$  values obtained in this TLC system were adequate for separation and identification of each compound.

#### REFERENCES

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