Table 1. Analysis of thioureas and halides in their mixtures

S. No.	Thiourea	Halide	Taken		Total wt. W (mg)	Found	
			thiourea (mg)	halide (mg)		thiourea (mg)	halide (mg)
1.	Thiourea	NaCl	21.0	55.0	76.0	21.186	54.814
2.	Phenylthiourea	KCl	19.8	42.0	61.8	19.920	41.880
3.	Methylthiourea	NH_4Cl	12.6	39.2	51.8	12.698	39.102
4.	n-Propylthiourea	NaBr	16.8	74.0	90.8	16.882	73.918
5.	p-Tolylthiourea	KBr	25.0	70.0	95.0	25.206	69.794
6.	Ethylthiourea	KJ	15.0	50.0	65.0	15.106	49.894
7.	p-Methoxyphenylthiourea	KJ	26.4	63.4	89.8	26.512	63.288

of the compound was dissolved in water and the volume made to 1 l. The solution was standardised by titration with 0.1 N silver nitrate using ferric alum as indicator.

Thiourea (GR, S. Merck) was used as such. *Phenylthiourea* (BDH) was purified by repeated crystallisation from ethanol until its m.p. was 154.0°C.

Other aryl and alkyl-thioureas, namely, o-tolylthiourea, p-tolylthiourea, o-methoxyphenylthiourea, p-methoxyphenylthiourea, o-ethoxyphenylthiourea, p-ethoxyphenylthiourea, methylthiourea, ethylthiourea, n-propylthiourea and allylthiourea were prepared by known methods [3, 6].

Ferric Alum Indicator. 1.0 g of ferric alum (BDH) was dissolved in 100 ml of water and 1—2 ml of conc. nitric acid was added to make the solution clear.

All other reagents were of chemically pure grade (BDH).

Procedure

A known weight (10—100 mg) of the mixture of thiourea and halide was dissolved in water or ethanol (about 10 ml) in a titration flask. To this a known excess of 0.1 N silver nitrate solution was added followed by 2 N ammonium hydroxide to keep the final concentration of the solution at 0.25—2 N with respect to ammonia. The flask was loosely stoppered and the contents were heated to boiling. The solution was cooled to room temperature and acidified with approximately 4 N nitric acid such that acidity ranged between 1—2 N with respect to the acid. The precipitated silver sulphide and silver halide were removed after filtration and thorough washing with water. The unreacted silver nitrate in the filtrate and washings was titrated against 0.1 N ammonium thiocyanate solution using ferric alum as indicator, to the appearance of pink colour.

The amounts of halide and thiourea in the mixture were calculated as:

$$\text{weight of halide (mg)} = \frac{M_1 M_2}{2M_1 - M_2}\left(\frac{2W}{M_2} - VN\right),$$

$$\text{weight of thiourea (mg)} = W - \frac{M_1 M_2}{2M_1 - M_2}\left(\frac{2W}{M_2} - VN\right),$$

where V denotes the volume in ml of N normal silver nitrate solution consumed by W mg of the mixture of halide and thiourea; and M_1, M_2 stand for the molecular weights of halide and thiourea, respectively.

Some typical results of the determinations are shown in Table 1.

The titrations were repeated with mixtures of halides with various alkyl and aryl-thioureas. Similar results as recorded in Table 1 were obtained.

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Berichtigung

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Unter „Arbeitsvorschrift" muß es heißen: „Schreiberempfindlichkeit 10 mV" (nicht 120 mV).