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Note

Gas chromatography of tributyltin(IV) N,N-dialkyldithiocarbamates

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Tributyltin(IV), N,N-dialkyldithiocarbamates, $SnBu_3$(DADTC), can be prepared from sodium N,N-dialkyldithiocarbamates and tributyltin(IV) chloride. They are viscous liquids, stable in alkaline solutions, but decompose in acidic solutions. Some properties of $SnBu_3$(DADTC) are similar to those of dithiocarbamates, others to those of tetraalkyltin(IV).

Since both tetraalkyltin(IV) and the dithiocarbamates of many metals can be analyzed by gas chromatography (GC), we have studied the basic conditions for GC analysis of $SnBu_3$(DADTC). The compounds separated are listed in Table I. In these analyses we exploited experience gained with the GC separation of dialkyldithiocarbamates of divalent metals[1–3].

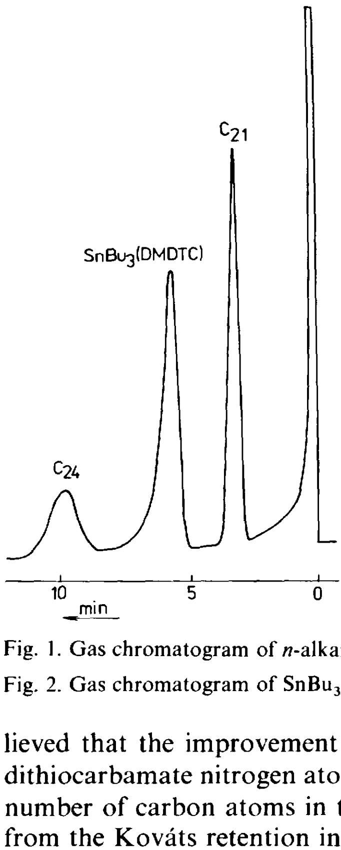
The tributyltin(IV) N,N-dialkyldithiocarbamates were separated in a glass column 0.55 m × 3.5 mm I.D.) packed with Chromaton N-Super (0.12–0.16 mm) (Lachema, Brno, Czechoslovakia) coated with 3% OV-101 polydimethylsiloxane (Supelco, Belefonte, PA, U.S.A.). A Fractovap 2300 gas chromatograph equipped with a flame ionization detector (Carlo Erba, Milan, Italy) was employed. Nitrogen was used as a carrier gas. The samples were injected as chloroform solutions using a 10-μl Hamilton microsyringe. *n*-Alkanes were used as reference materials.

Fig. 1 shows a chromatogram of a mixture of *n*-alkanes (C_{21} and C_{24}) and $SnBu_3$(DMDTC). It is seen that the peak of $SnBu_3$(DMDTC) is not symmetrical. A chromatogram of $SnBu_3$(DEDTC) is shown in Fig. 2. The peak is more symmetrical than those in Fig. 1, *i.e.*, peak symmetry is improved by increasing chain length of the alkyls in the dithiocarbamate ligand (methyl to ethyl). We have observed a similar effect in the GC separation of dialkyldithiocarbamates of divalent metals[3]. It is be-

TABLE I

LIST OF COMPOUNDS ANALYZED

Compound	*Abbreviation*	*M.p. (°K)*
Tributyltin(IV) N,N-dimethyldithiocarbamate	$SnBu_3$(DMDTC)	437
Tributyltin(IV) N,N-diethyldithiocarbamate	$SnBu_3$(DEDTC)	449
Tributyltin(IV) N,N-dipropyldithiocarbamate	$SnBu_3$(DPDTC)	457
Tributyltin(IV) N,N-dibutyldithiocarbamate	$SnBu_3$(DBDTC)	463



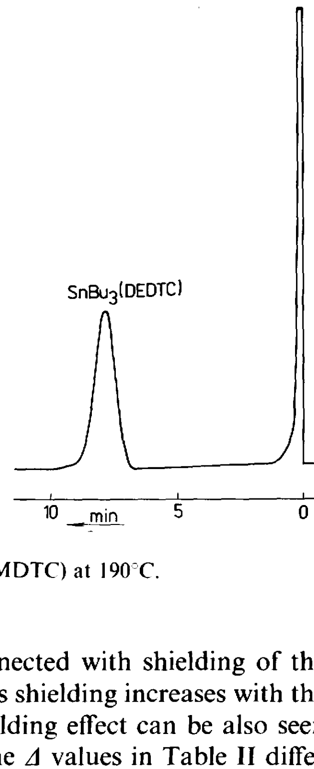


Fig. 1. Gas chromatogram of *n*-alkanes (C_{21} and C_{24}) and $SnBu_3$(DMDTC) at 190°C.

Fig. 2. Gas chromatogram of $SnBu_3$(DEDTC) at 190°C.

lieved that the improvement of peak symmetry is connected with shielding of the dithiocarbamate nitrogen atom by the alkyl groups. This shielding increases with the number of carbon atoms in the alkyl groups. This shielding effect can be also seen from the Kováts retention indices listed in Table II. The Δ values in Table II differ from the theoretical ones; an increase in the number of carbon atoms as in methyl and diethyl should correspond to 200 index units. Moreover, the Δ values increase with increasing carbon number, as was found for dialkyldithiocarbamates of divalent metals[3].

TABLE II

KOVÁTS RETENTION INDICES OF TRIBUTYLTIN(IV) DIALKYLDITHIOCARBAMATES AT 463°K

Compound	*I*	Δ
$SnBu_3$(DMDTC)	2251	
$SnBu_3$(DEDTC)	2303	52
$SnBu_3$(DPDTC)	2415	112
$SnBu_3$(DBDTC)	2566	151

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