

SHORT COMMUNICATIONS

Gas Chromatography of Rare Earth Chelates of Pivaloyltrifluoroacetone

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The gas chromatography of rare earth chelates of a new β -diketone, pivaloyltrifluoroacetone (PTA) was investigated. The trifluoromethyl group enhances the volatility of these chelates and the *t*-butyl one shows the shielding effect to weaken the intermolecular bonding. By these effects, rare earth PTA chelates were eluted in the gas phase as well as dipivaloylmethane¹⁾ and 1,1,1,2,2,3,3-heptafluoro-7,7-dimethyl-4,6-octanedione (fod)-chelates.²⁾

PTA was synthesized by condensation of ethyl trifluoroacetate with pinacolin with sodium methoxide. The crude product was purified by con-

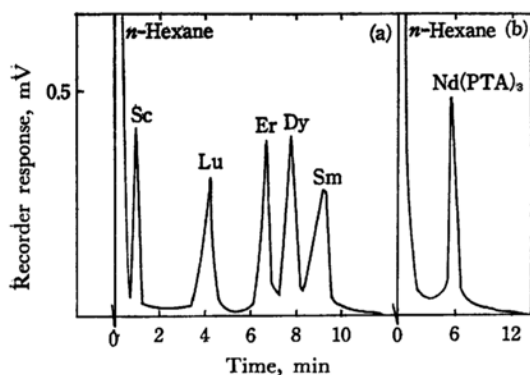


Fig. 1. Chromatograms of some rare earth PTA chelates.

- a) The separation of Sc(PTA)_3 , Lu(PTA)_3 , Er(PTA)_3 , Dy(PTA)_3 and Sm(PTA)_3 . Column temp.: programmed from 150 to 260°C at 12°/min. Injection port temp.: 280°C. Helium flow rate: 50 ml/min.
- b) Chromatogram of Nd(PTA)_3 . Column temp.: programmed from 200 to 270°C at 12°/min. Injection port temp.: 320°C. Helium flow rate: 43 ml/min.

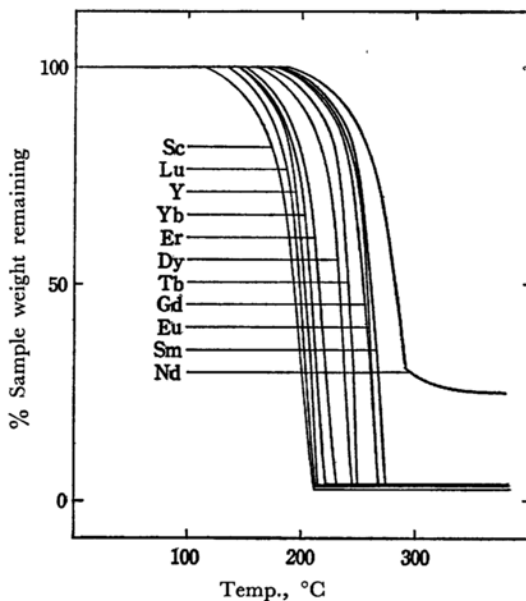


Fig. 2. Thermogravimetric curves of rare earth PTA chelates.

verting it into the copper chelate, which was dissolved in ether, decomposed in 10% sulfuric acid, and distilled (bp 39–41°C/17 mmHg). Tris PTA chelates of the following rare earth elements were prepared; Sc, Y, Lu, Yb, Er, Dy, Tb, Gd, Eu, Sm and Nd. The column (75 cm \times 3 mm i. d. stainless steel) was packed with 5% (W/W) silicone grease on 60–80 mesh Chromosorb W. The chromatograms of these metal chelates were obtained, of which a typical example was shown in Fig. 1. The order of elution is the same with that of increasing ionic radius of the central metal. The thermogravimetric curves are shown in Fig. 2. They approach 100% weight loss of the chelates, except the case of Nd(PTA)_3 . Figure 2 indicates that the volatility of the chelate increases with decrease in the ionic radius of rare earth metal ion.

1) K. J. Eisentraut and R. E. Sievers, *J. Am. Chem. Soc.*, **87**, 5254 (1965).

2) C. S. Springer, Jr. D. W. Meek and R. E. Sievers, *Inorg. Chem.*, **6**, 1105 (1967).