

After removing the excess hydrochloric acid under vacuum, the residue which contained cystine was dissolved in warm water and filtered. The filtrate was diluted to 50 ml, from which 25 ml solution was taken and followed the procedure as outlined for cystine.

Results and Discussion

The results obtained in the analysis of cystine are accurate within 0.8%. The comparison of the results obtained in the proposed method and paper chromatography procedure² showed that they agree within 2% error. Beer's law is found to be valid over the concentration range 0.001 to 0.0075 millimoles of cystine per 25 ml with molar absorptivity value 1.73×10^3 l. mole⁻¹ cm⁻¹.

There is no interference from alanine, glycine, valine, leucine, lysine, tryptophan, serine, threonine, isoleucine, glutamic acid, aspartic acid, arginine, tyrosine, phenylalanine, methionine, cystine, glucose, lactose, sucrose, purines and pyrimidines upto 2 millimoles but histidine, proline and hydroxyproline interfere when present in concentrations more than one millimole. However, glutathione interferes. The method is found to be unsuitable in presence of ascorbic acid, epinephrine and heavy metals such as mercury and lead. Hence, the reaction appears to be highly specific for cystine and has the advantage that cystine need not be separated from the amino acid mixtures or from the remaining amino acids which are present in protein hydrolysates.

The authors (RRK and PS) are grateful to the authorities of Andhra University and to C.S.I.R. (India), for the award of research fellowships.

Department of Chemistry, R. RAMA KRISHNA,
Andhra University, P. SIRAJ,
Waltair 530 003, C. S. PRAKASA SASTRY.
June 26, 1979.

1. Subba Rao, D. and Hanumantha Rao, K., *The Ind. J. Nutr. Dietet.*, 1972, 9, 331.
2. Hanumantha Rao, K. and Subrahmanian, N., *J. Food Sci. Technol.*, 1970, 7, 31.

CATIONS OF TETRAPHENOXY NIOBIUM(V) AND TANTALUM(V) CHLORIDES

NIOBIUM(V), tantalum(V) and antimony(V) chlorides act predominantly as chloride ion acceptors¹⁻³. The spectra of addition compounds of tetrachlorides of sulphur, selenium and tellurium with niobium(V) and tantalum(V) chlorides show the presence of pyramidal MCl_3^+ and weakly perturbed octahedral $NbCl_6^-$ and $TaCl_6^-$ ions in the crystal⁴. Raman spectra of solidified $SbCl_5$ - $NbCl_5$ mixtures have been explained on the basis of a dimer $SbNbCl_{10}$.⁵ Cationic complexes of

the type T_4Nb^+ and T_4Ta^+ where T is the tropolene cation have been stabilized by large symmetrical anions.⁶⁻⁸ An attempt has therefore been made to explore the possibility of the formation of ions of the type $M(OC_6H_5)_4^+$ from these phenoxides.

Tetraphenoxy niobium(V) and tantalum(V) chlorides were prepared by the methods as described in literature^{9,10}. Compounds of composition $MSb(OC_6H_5)_4Cl_6$ were prepared by mixing the two components in 1:1 molar ratio in dichloromethane. The resulting solutions were refluxed for one hour, when reactants went into solution. The compounds were isolated by the addition of inert solvents like petroleum ether. They were filtered in dry atmosphere and finally dried under vacuum.

Stoichiometric composition of these compounds has been established by carrying out conductometric titrations of tetraphenoxy niobium(V) and tantalum(V) chlorides versus antimony(V) chloride; in nitrobenzene at $25 \pm 0.1^\circ C$. The conductance-Composition curve (Fig. 1) reveal sharp breaks at 1:1 molar ratio suggesting that the resulting compound has 1:1 stoichiometry. The continuous increase in conductance of the solution has been attributed to the formation of ions in solution, thus excluding the possibility of the formation of non conducting compounds of the type $SbNbCl_6(OC_6H_5)_4$. These compounds are crystalline solids and have high melting points. They are insoluble in nitromethane, acetonitrile but fairly soluble in nitrobenzene. Molar conductance values of millimolar solutions of these compounds in nitrobenzene suggest their ionic character.

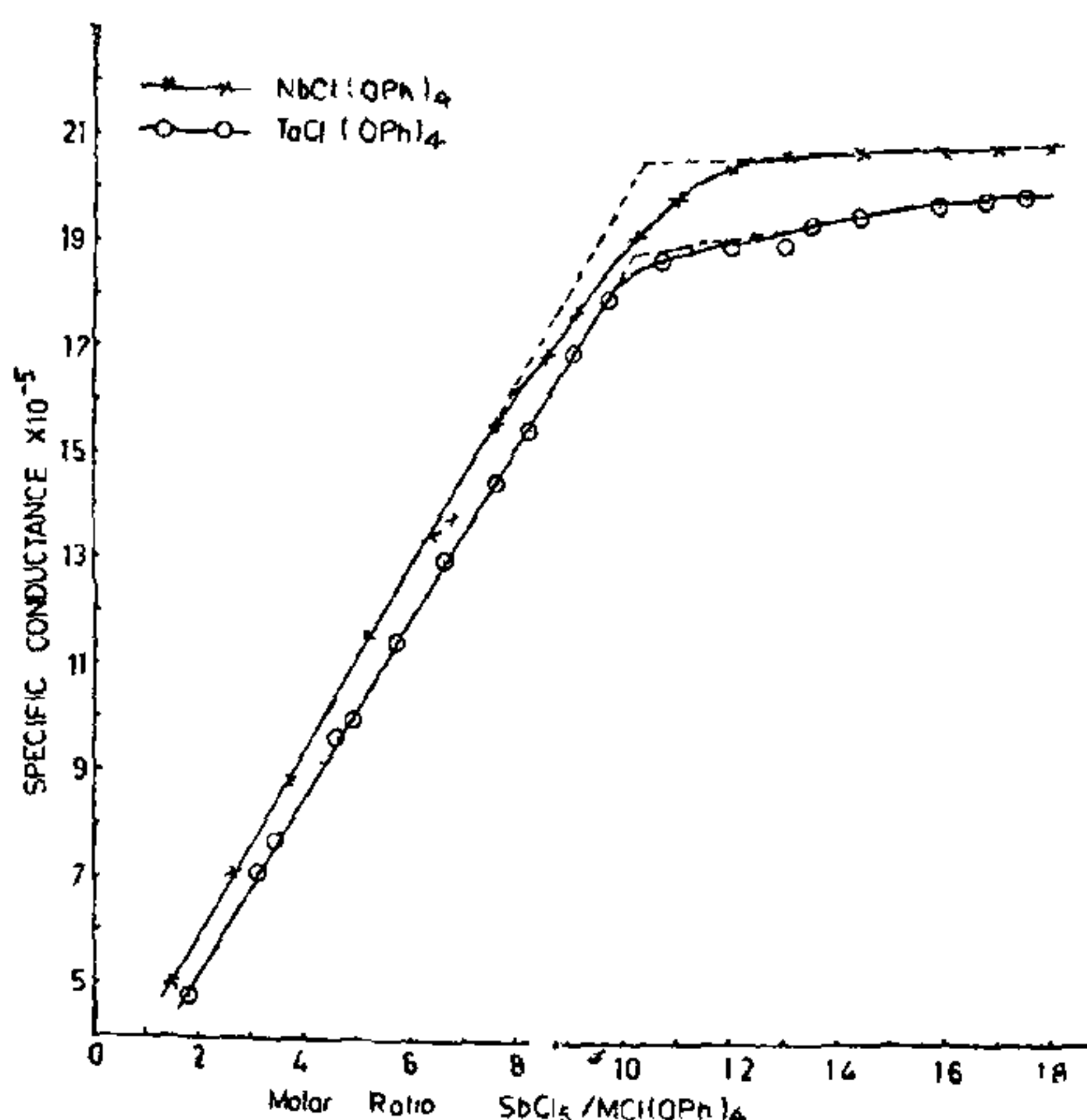
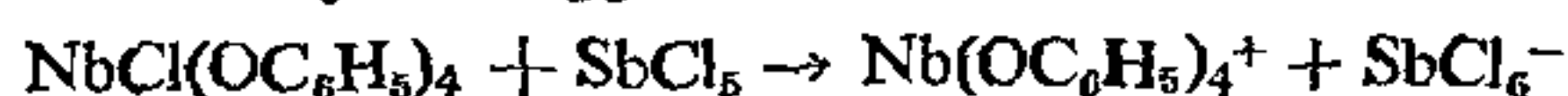


FIG. 1. Conductometric titrations of tetraphenoxy M(V) chloride against $SbCl_5$ in nitrobenzene at $25 \pm 0.1^\circ C$.

Information on the structure of these compounds has been obtained from their infrared spectra. Very sharp bands around 620 and 338 cm^{-1} are characteristic of $\nu(\text{Sb}-\text{Cl})$ stretching modes in octahedral environment¹¹. The band at 348 cm^{-1} present in tetraphenoxy niobium(V) chloride due to terminal $\nu(\text{Nb}-\text{Cl})$ has been found to be completely missing which suggest that chlorine of the phenoxy derivative has been transferred to antimony making octahedral environment around it. Besides this, a lowering of about 8-10 cm^{-1} has been observed in (M-O) stretching modes of the phenoxy derivative on complexation with antimony(V) chloride. Apart from these important bands, the other bands present at 1596, 1498, 1472, 1384, 1182, 1167, 884, 825, 688 cm^{-1} assigned to $\nu(\text{C}=\text{C})$, $\nu(=\text{CH})$, $\nu(\text{C}-\text{C})$ ring present in phenol¹² shift to lower spectral regions suggesting the presence of positive charge on the metal surrounded by phenoxy groups. These observations suggest chlorine abstraction from tetraphenoxy niobium(V) and tantalum(V) chloride by antimony(V) chloride, thus stabilizing the cations $\text{M}(\text{OC}_6\text{H}_5)_4^+$ by large symmetrical anion SbCl_6^- which is in agreement with earlier observations⁸. A possible reaction may be suggested as:



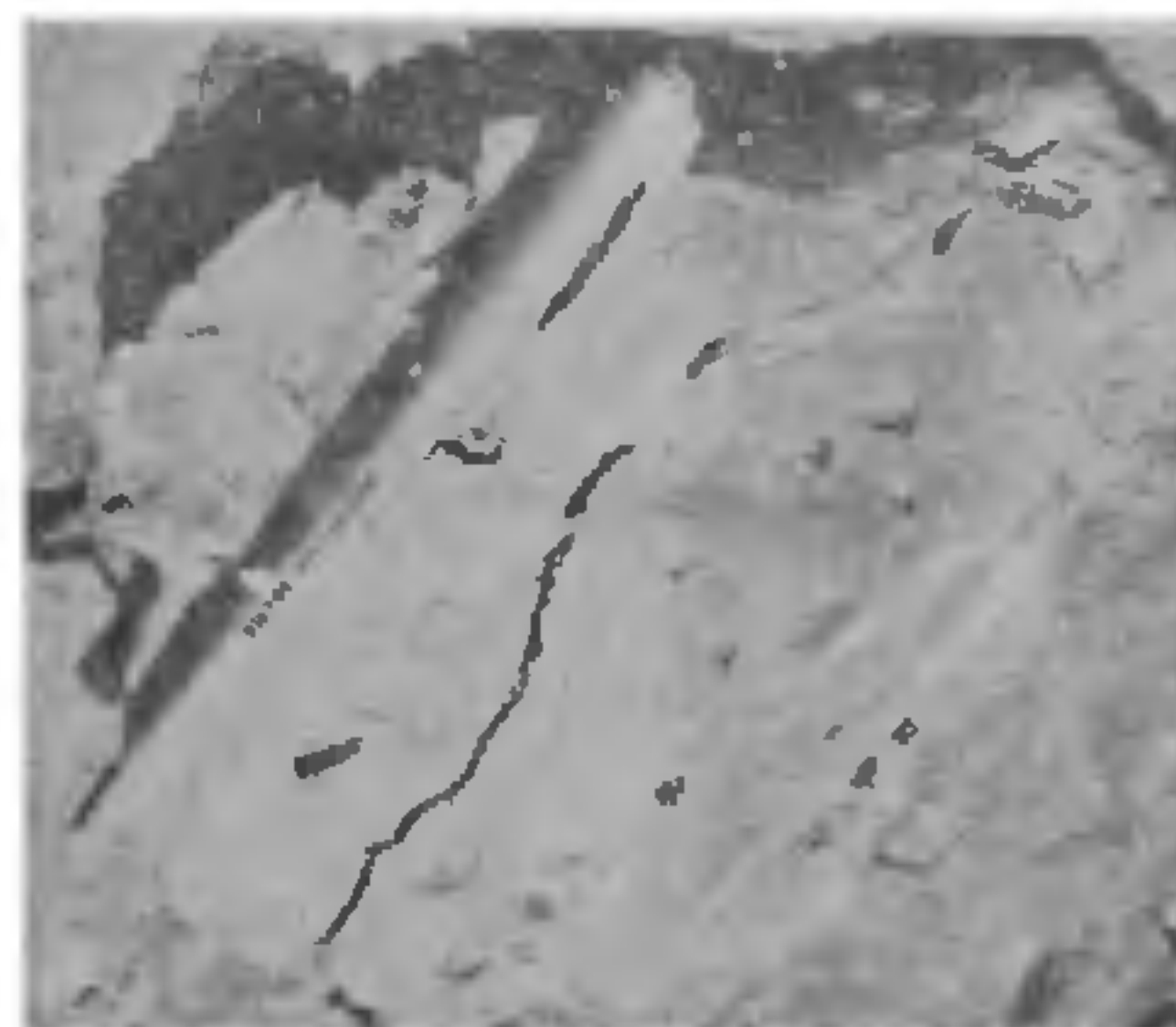
One of us (UKB) is thankful to Himachal Pradesh University, for the award of a junior research fellowship.

Chemistry Department,	K. C. MALHOTRA.
Himachal Pradesh University,	U. K. BANERJEE.
Simla 171 005, June 25, 1979.	S. C. CHAUDHRY.

1. Gutmann, V. and Mairinger, F., *Z. anorg. allgem. Chem.*, 1957, 289, 279.
2. Kolditz, L., *Halogen Chemistry* (ed. V. Gutmann), Vol. 2, Academic Press, New York, 1967.
3. Beveridge, A. D. and Clark, H. C., *Halogen Chemistry* (ed. V. Gutmann), Vol. 3, Academic Press, New York, 1967.
4. Poulsen, F. W. and Berg, R. W., *J. Inorg. Nucl. Chem.*, 1978, 40, 471.
5. Bues, W., Demiray, F. and Brockner, W., *Spectrochim. Acta*, 1976, 32A, 1623.
6. Muttarties, E. L. and Wright, C. M., *J. Am. Chem. Soc.*, 1965, 87, 21.
7. — and —, *Ibid.*, 1965, 87, 5706.
8. Fairbrother, F., *Chemistry of Niobium and Tantalum*, Elsevier Publishing Company, New York, 1967, p. 58.
9. Prakash, S. and Kapoor, R. N., *Ind. J. Chem.*, 1967, 5, 50.
10. Schoenherr, M., Hass, D. and Baufeld, K., *Z. Chem.*, 1975, 15, 66.
11. Cook, D., Kuhn, S. J. and Olah, G. A., *J. Chem. Phys.*, 1960, 33, 1669.
12. Green, J. H. S., *Ibid.*, 1961, p. 2236.

A REPORT ON THE OCCURRENCE OF GYROCHORTE AND OTHER BILOBED TRACE FOSSILS FROM THE JAISALMER FORMATION, RAJASTHAN

A WELL PRESERVED ichnofaunal horizon is being reported from Bada Bagh near Jaisalmer in Rajasthan. The horizon contains *Gyrochorte* and other bilobed traces which belong to the Bada Bagh member of Jaisalmer Formation (Callovo-Oxfordian).¹ These fossils are found in yellow, fine grained sandstones which form the uppermost unit of the member, and are 50 ft. thick. This unit also contains small brachiopods, echinoids, corals, and bryozoans.



Gyrochorte



Bilobed trace fossil

The ichnogenus *Gyrochorte* Heer, 1865 is a bilobed crawling trace occurring as positive epirelief over the rock surface. The lobes are biserially arranged with a median furrow. These are 1 to 2 mm high, 4 to 5 mm wide, unbranched, and are freely wound cutting across each other. The surface of each lobe is marked by a transverse furrow which meets the median furrow at about 35° angle. These transverse furrows are almost 3 mm apart and appear alternating on each lobe. Hallam² has discussed in detail about its possible mode of origin, direction of movement of animal concerned, and its palaeoecological significance. The other bilobed traces are smooth with a median furrow and are much more abundant than *Gyrochorte*. These traces are 3 to 6 mm wide, 1 mm high, are freely winding and cutting