

R.A. Lidgett, Ruabon

13th April, 1972

Aroclor 1242 Analysis

RAL:eh

E.M. Emery, St. Louis

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TIB

Many thanks for your recent telexes and comments. We are holding issue of any method for determination of penta Cl and components in 1242 until MCL and MC agree on all aspects of the analysis.

Receipt of your last telex prompted us to re-examine our g.c./m.s. work and has resulted in a number of observations which I am sure are important in both the method development side and in possible plant control.

G.C./M.S. Peak Identification

Tarry Gay did identify peak 30 as containing a mainly 5Cl component but has not detected significant amounts in the other peaks particularly peak 31 which he allocates to Cl4.

However, when he examined 1248 there was a definite increase in Cl5 components in the peaks mentioned by you and, more significantly, I think, Cl5 peaks appeared at shorter retention times than have been detected in 1242. While we accept that this analysis is prone to difficulties and it is possible that we have missed Cl5 in some of the small peaks in 1242 we think that there is a good possibility that the composition of 1242 is very dependent upon chlorination level and that slight increases in chlorine content may affect the Cl5+ concentration more than we might have expected. This is at present supposition but could explain differences in MC and MCL results and in the differences found between various batches of MCL material. We are at present examining a range of samples from Newport to determine if this is in fact the case.

We will also be discussing this with Newport TSD to see whether examination of samples at the low and high ends of the 1242 specification can be carried out.

Because of the critical effect that g.c./m.s. has on the determination of composition of 1242 we have also arranged to meet the Unilever people to discuss the general difficulties associated with P.C.B. analysis. Examination of their mass spectra may help to clear up peak identification differences.

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Absolute Level of Penta Cl<sub>4</sub> Components in 1242 sent to Prodelec

As discussed above it may not be possible to analyse a 1242 sample by allocating Chlorine numbers to the various peaks and assuming that these do not change composition with small changes in chlorination level. However, in the sample sent to Prodelec I do not think that the effect of peaks 30, 32 and 38 will greatly change the result since apart from peak 38 these are present in 1242B which has a very low Cl<sub>15</sub><sup>+</sup> content.

Peak 31 remains a problem since if it does contain 25% Cl<sub>15</sub> then I agree it will increase the total Cl<sub>15</sub><sup>+</sup> concentration significantly. Perhaps this anomaly will be clarified when you have looked at the sample of Prodelec material - if Cl<sub>15</sub> is present in that sample we will have to reassess our g.c./m.s. results.

Further Developments

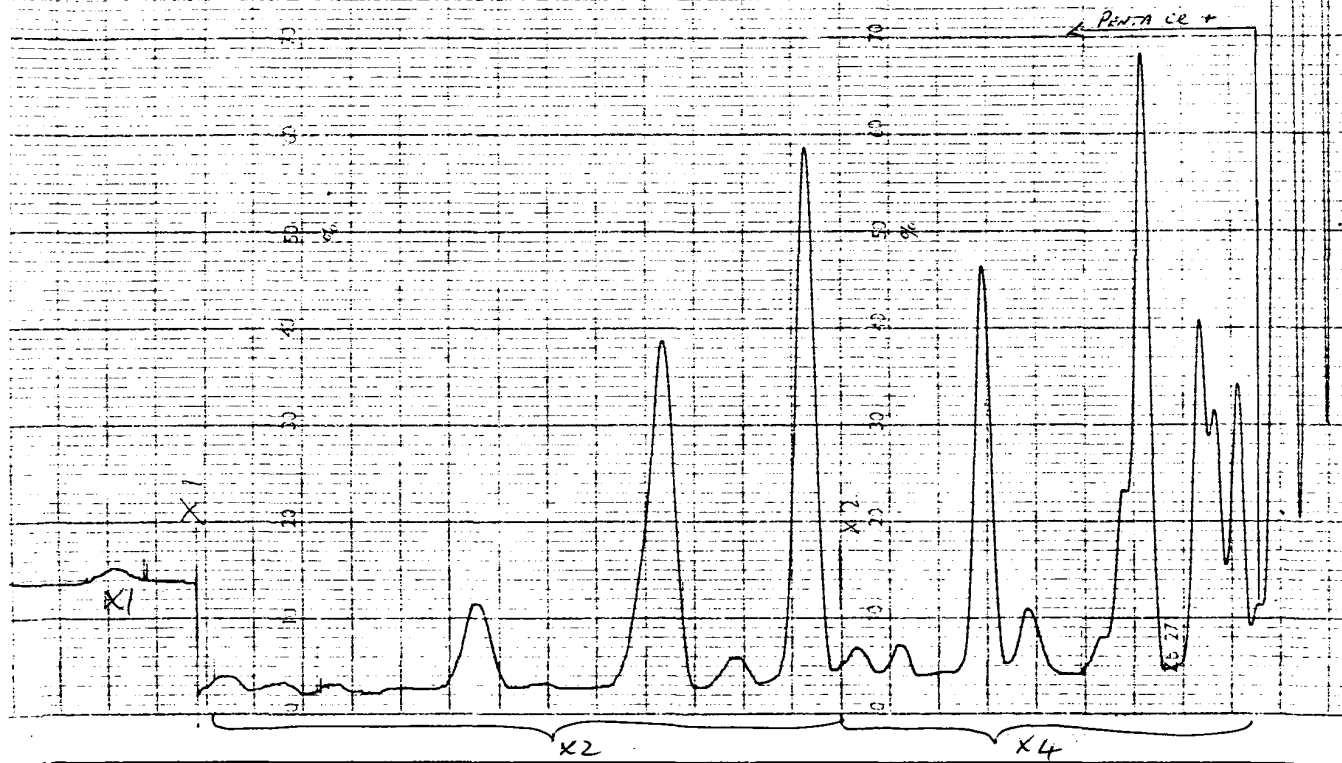
In the last few days we have been testing a 300' x 64 thou. S/S Apizon L column and results are promising (chromatogram attached). If stable we will be able to use this column without split and with a loading of 1-3µl of sample. We should therefore increase sensitivity to high boilers and for m.s. applications. I will let you know what happens.

  
R.A. Lidgett

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5  $\mu$ l PRODRELIC SAMPLE

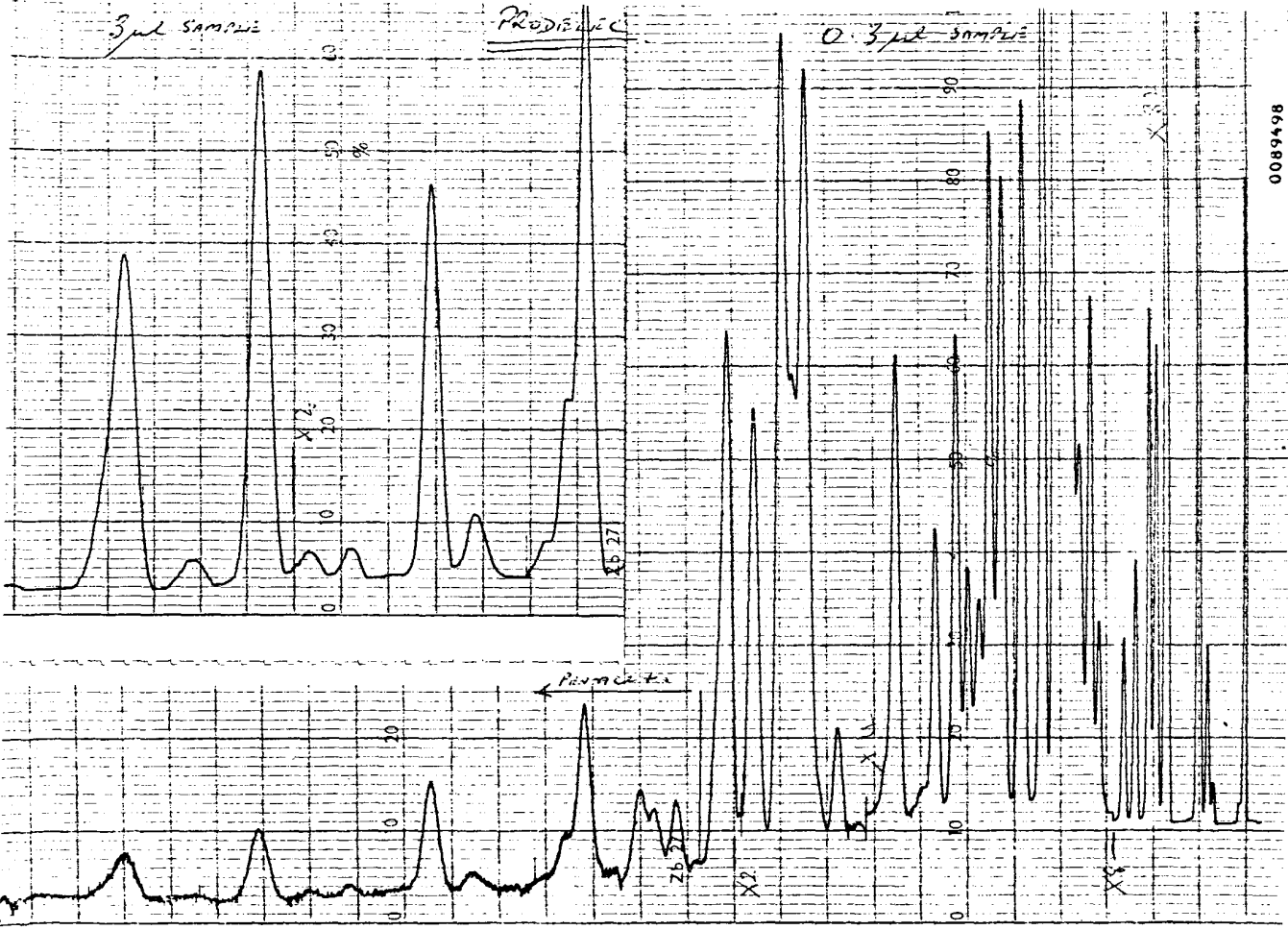
300' 64 TAU APPARATUS



3  $\mu$ l sample

PRODISEC

0.3  $\mu$ l sample



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