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Journal of Fluorine Chemistry 71 (1995) 219–220

JOURNAL OF FLUORINE CHEMISTRY

Use of co-distillation of reactive fluorine compounds in spectroscopic studies and trace gas analysis

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Keywords: Co-distillation; Reactive fluorine compounds; IR spectroscopy; Matrix isolation; Trace gas analysis

Fractional co-distillation, first described by Cady and Siegwarth [1], is the most gentle method of separation for volatile, reactive compounds. With the exception of the separation column, which is exchanged by a 'Cady tube' [2], the apparatus employed is in general identical to that used in gas chromatography.

If a Cady tube is coupled with a matrix isolation device, the carrier gas (Ne, Ar, N_2, etc.) can be quenched as a matrix and detection of the matrix-isolated species is possible by infrared spectroscopy. A typical experimental set-up is shown in Fig. 1. In order to prepare a matrix, the compound of interest, trapped in the Cady tube, is warmed until its vapour pressure increases to ca. 10^{-3} mbar. The temperature must then be kept constant while the vapour is swept out with a constant stream of matrix gas which is subsequently quenched on the matrix support. In this manner we have been able to prepare matrices containing CrF_5 [3] and SF_3CN [4] without any detectable impurities.

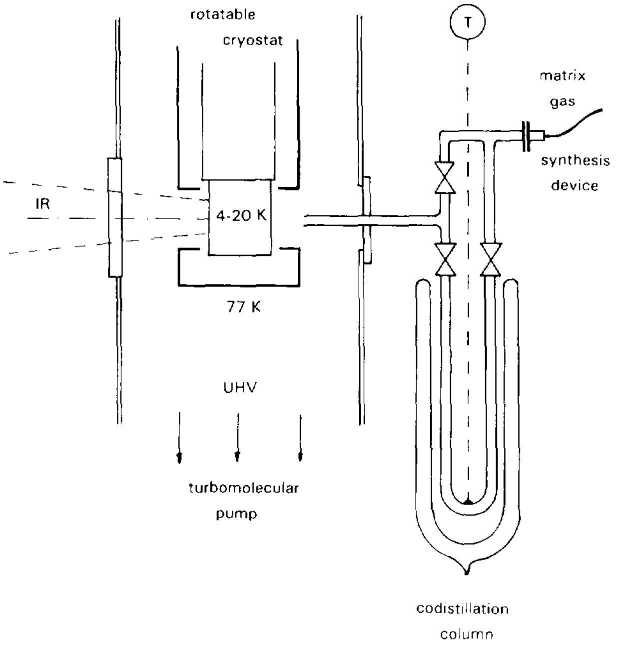

Fig. 1. Typical experimental set-up for separation of volatile, reactive compounds by co-distillation.

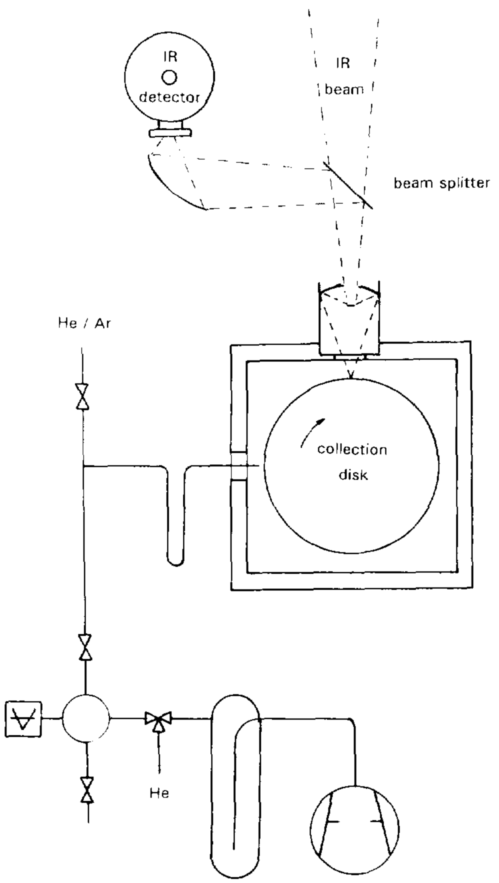

Fig. 2. Matrix isolation of very reactive compounds.

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SSDI 0022-1139(94)06033-9

Very recently it has become possible to scale down the size of the Cady tube, so that trace gas analysis of reactive species is possible [5]. For this purpose, we have used stainless steel or quartz glass capillaries as Cady tubes coupled with a specially designed matrix-isolation apparatus [6] (see Fig. 2). This operates in a similar manner to the commercial cryolect® collector [7]. Mixtures of NOCl, $COCl_2$, SO_2, etc. have been separated and detected by infrared spectroscopy on a nanogram scale. Gram Smith traces look similar to ordinary co-distillation diagrams [5].

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