A device for centering samples in ESR measurement

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This simple device, designed for use with a Varian ED ESR spectrometer using 2 mm ID quartz tubes, is easy and cheap to construct, efficient to use and improves reproducibility for both mass and density normalized aliquots. Because much of my work has been on alpha irradiated samples, the sample size for ESR cavity is particularly significant for these small samples, which occupy only a few mm of the cavity window, and accurate positioning, both vertically and horizontally, is absolutely critical.

The sample tube holder shown in figure 1 ensures the sample is held vertical: the usual holder for the bottom of the tube produces too much noise and distortion if it is introduced into the cavity. Another device, also shown in figure 1, allows accurate, fast positioning of the sample in the vertical centre of the cavity. A standard sample is centred by trial and error to maximize the ESR intensity, the tube and sample holder transferred to the centering device and the depth of insertion set. The next sample tube can then be inserted to the same depth before being transferred to the spectrometer. This greatly improves reproducibility, as well as reducing the time taken for each changeover and hence the effect of machine drift (a real problem with this particular instrument) to acceptable levels.

Experimentation shows that for small samples, accurate positioning alone is not sufficient for good reproducibility, as the degree of packing can give rise to a variation of up to 15% in the ESR intensity value even for correctly positioned samples. Calculations based on the way the cavity varies in sensitivity and assuming a potential variation in packing density of 30%, confirms these data, and it is important that mass normalized samples, even when correctly positioned, be packed to a uniform height. The same care to ensure uniform packing is needed when more sample is available and the common practice of overfilling the sample tubes to ensure that the cavity window (10 mm height) is completely covered can be adopted. A simple correction based on average density, may still leave significant uncertainties (up to 10%) in the ESR intensity, unless the sample has constant density.

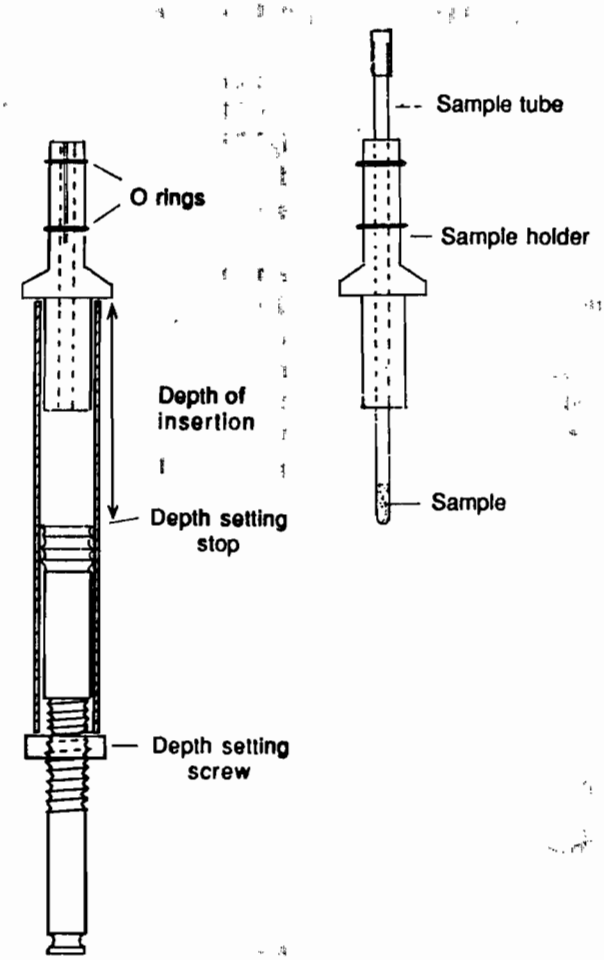

Figure 1.
The sample holder and depth setting device. The sample tube is held vertically and tensioned by 2 rubber rings so that no insert is needed in the cavity for centring. The sample tube in its holder is inserted in the device which is set to a predetermined depth to give maximum sensitivity for the sample height. The device is constructed of teflon with a glass cylinder for visual checking.

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This is a practical device for a specific model of spectrometer. Similar devices could be constructed for other spectrometers and would be useful for assuring minimum error is ESR signals. The packing of the powder in the tube is also important, as noted; the effect of variable packing can be controlled by repeatedly tapping each tube on a hard surface, after the sample has been loaded and before insertion into the holder.