

Sample preparation ②

Product used : Electron Spin Resonance (ESR)

■ Sampling of solution sample

Solution samples may be subject to restrictions on the volume of samples and the shape of the sample tube due to the dielectric loss of the solvent. Dielectric loss is a phenomenon in which a portion of the energy is lost as heat within the dielectric when an alternating electric field is applied to it. ESR measurement of a sample with dielectric loss can result in decreased measurement sensitivity due to this effect. It is necessary to select a sample tube suitable for the specific solvent, as the dielectric loss varies depending on the solvent type.

When observing specific radicals in solution samples, it is recommended to use solvents with low dielectric loss. If a solvent with significant dielectric absorption is used, it is essential to minimize the measurement sensitivity loss by properly selecting the sample tube. For more information about the solvent effect, please refer to the application note ER220006E.

■ Example of ESR measurement – aqueous solution of TEMPOL

We present examples of ESR measurement using TEMPOL, which is diluted with ultrapure water to the same concentration, with various sample tubes. The volumes and inner diameters of the collected sample tubes are listed in Table 1. The aqueous sample cell (ES-LC12) specifications indicate the inner diameter of the short side. Capillary tubes were inserted into a standard sample tube for ESR measurement. Figure 2 shows the sample volume and the integration value of the observed ESR signal.

As depicted in Figure 2, in ESR measurement using ①, the ESR signal was barely detectable due to the decreased measurement sensitivity caused by the effect of dielectric loss. ESR measurement sensitivity is reduced for water solution samples with a large dielectric loss, using the standard sample tube with an outer diameter of 5 mm for ESR measurement of water solution samples would be difficult. However, ESR measurement with ② maintained sensitivity by mitigating the impact of dielectric loss and ensuring sufficient sample volume. The ESR measurement sensitivity is influenced by the dielectric loss of the solution sample, which is affected by the solvent and the inner diameter of the sample tube. The dielectric loss of the water solution becomes more pronounced for larger inner diameters of the sample tube or capillary tubes.

Table 1. Specification of sample tubes

Symbol	Sample tube	Inner diameter (mm)	Volume (μL)
①	Standard sample tube	4	10
②	Aqueous sample cell	0.25	130
③	Capillary tube	0.47	8
④	Capillary tube	1	35
⑤	Capillary tube	1.3	59



Figure 1. ESR spectrum of TEMPOL solution

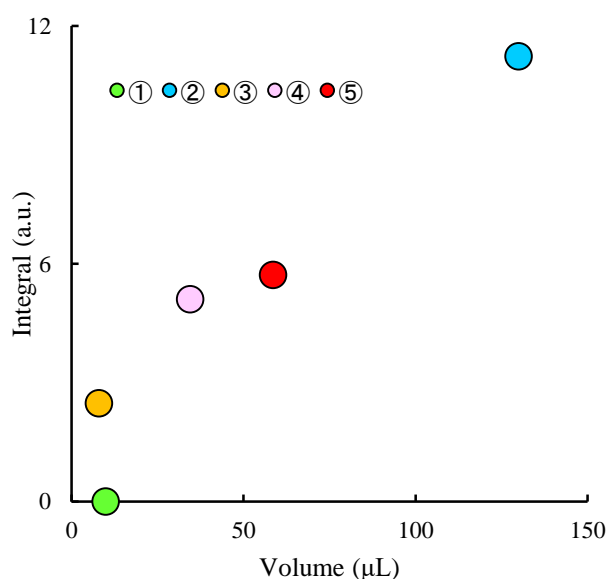


Figure 2. Volume and integration value of samples

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