



Annex IV

Instructions for recovery experiments and reporting sheet

This test must be performed by the applicants exactly as given in this instruction as it constitutes the initial basis of comparison between different markers used to short-list four candidate markers that will undergo further tests. Documentary evidence of the quality of the fuels used and a statement about the purity of the marker is required in addition to the analytical results.

1: Request a kit of 8 silica cartridges from EC-JRC-IRMM (address in the call under 4.2)¹

2: Prepare 4 x 500 mL of marked fuel at the suitable marking level in two different gas oil qualities. The appropriate marking level must be chosen by the supplier.

3. Out of these four solutions prepare 2 x 500 mL of fuel in regular low sulfur gas oil that fulfils diesel EN590. The appropriate marking level must be chosen by the supplier.

4. The remaining 2 x 500 mL solutions must be prepared in B7 gas oil using biodiesel fulfilling diesel EN14214. The fraction of biodiesel in the regular gas oil must be 7 ± 1 % (v/v). The final B7 gas oil must fulfil diesel EN590. The appropriate marking level must be chosen by the supplier.

5: Measure the concentration of marker in each of the freshly prepared fuel samples with the analytical technique and method suitable to measure the particular marker and report the results in mg/kg. The analytical method and instrumentation is up to the supplier to choose.

6: Condition the silica cartridges by passing 10 mL of hexane and let run dry.

7: The cartridges should be placed in a holder and the experiment should be done at ambient pressure letting the sample pass through by gravity. Perform four replicate recovery-tests by passing portions of the marked fuels through the silica cartridges with a flow-rate of 5 ml per minute and collect and measure each fraction separately. If 5 ml per minute cannot be achieved the flow rate must be recorded and reported. Please collect the volumes as indicated in the table below.

¹ It is the responsibility of the applicant to request the cartridges from JRC on time taking into consideration the time necessary to process the request (maximum five working days) and for delivery.

8: Measure the concentration of marker in each fraction that has passed through the cartridge using the analytical technique and method suitable to measure the particular marker and report the result in mg/kg.

9: Report the results in the Table below, the breakthrough volumes and marking levels.

Table for reporting of marker concentration as a function of volume after passing over a silica cartridge of 35 mm length and 20 mm diameter.

	Silica cartridge 1	Silica cartridge 2	Silica cartridge 3	Silica cartridge 4
	Marked fuel solution 1	Marked fuel solution 2	Marked fuel solution 3	Marked fuel solution 4
	Regular gas oil fulfilling diesel EN590	Regular gas oil fulfilling diesel EN590	B7 gas oil fulfilling diesel EN14214 and EN590	B7 gas oil fulfilling diesel EN14214 and EN590
	Concentration of marker in each fraction, mg/kg			
20 mL				
40 mL				
60 mL				
80 mL				
100 mL				
140 mL				
180 mL				
220 mL				
260 mL				
300 mL				
400 mL				
500 mL				

1. Report the initial marking level in diesel EN590: _____ mg/kg
2. Report the initial marking level in B7 gas oil: _____ mg/kg
3. Report the breakthrough volume (at which volume the level of marker corresponds to 95 % of the original marking level) in diesel EN590:
_____ mL
4. Report the breakthrough volume (at which volume the level of marker corresponds to 95 % of the original marking level) in B7 gas oil:
_____ mL