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**15 October 2017**

**Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants  
Subcommittee D02.03 on Elemental Analysis**

**Research Report: D02-1875**

**Interlaboratory Study to Establish Precision Statements for ASTM  
D4929, Standard Test Method for Determination of Organic Chloride  
Content in Crude Oil, Procedure C, X-ray Fluorescence Spectrometry**

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1. **Introduction:**

The purpose of this ILS is to add Procedure C for X-ray Fluorescence Spectrometry to D4929. This ILS program number is 1122 and is associated with WK46925 and WK57076. This ILS was run in tandem with ILS 1329 (WK53424 and WK59006), which is a proposed method for organic chlorides in crude by distillation and Combustion Ion Chromatography (CIC). ILS 1329 results can be found in RR: D02-1868.

2. **Test Method:**

The Test Method used for this ILS is D4929-15a. When these Procedure C revisions went to D02.03 ballot and D02 ballot in 2017, D4929-16 was the active standard. To obtain a copy of D4929, go to ASTM's website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service by phone at **610-832-9585** (8:30 a.m. - 4:30 p.m. Eastern U.S. Standard Time, Monday through Friday) or by email at [service@astm.org](mailto:service@astm.org).

3. **Participating Laboratories:**

The following laboratories participated in this interlaboratory study:

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#### 4. Description of Samples:

The sample design for this ILS was intended to replicate samples used in the original D4929 Procedure A/Procedure B ILS (RR: D02-1293) as much as possible. Since the original study is dated December 1987, it was not possible to obtain identical crude oils. Assistance was requested from Dennis Sutton, Executive Director of the Crude Oil Quality Association, to identify similar crude oils for use in this study. Based on his suggestions, the following crudes oils were used for this ILS.

Crudes Used in RR:D02-1293 Study:	Crudes Used in This ILS:
Liberty, Miss.	LLS (Light Louisiana Sweet)
Butte, Mont.	Mars
West Texas	WTI (West Texas Intermediate)

LLS, Mars, and WTI bulk crude samples were provided by Marathon Petroleum.

Ten samples of varying targeted results used for this study. Each sample was prepared and doped according to the original study (RR:D02-1293) then packaged in quart containers in blind duplicate. The organic chloride dopants used in this study were identical to the dopants in the original study, except that 1,1,2-trichloroethane was used in place of 1,1,1-trichloroethane. Sample preparation and distribution were performed by Spectrum Quality Standards. Below is a list of the samples, dopant used, and doped chlorine concentration. (Note: The doped chlorine concentration does not take in account any chlorine that may already be in the bulk crude oil samples.)

Sample ID	Crude Oil	Organic Chloride Dopant	Doped Chlorine Conc (mg/kg)
A	LLS	1,1,2-trichloroethane	1
B	Mars	1,2-dichloropropane	12
C	Mars/LLS	1,1,2-trichloroethane	10
D	Mars	1,2-dichloropropane	3
E	WTI	Blank	0
F	LLS	1,2-dichloropropane	1
G	Mars	o-dichlorobenzene	5
H	LLS	Blank	0
I	WTI/Mars	methylene chloride	6