

SULFUR TEST METHOD PANEL

DIESEL WORKSHOP
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CHRONOLOGY

- **Final Highway Diesel Rule published in Federal Register in January 2001**
- **Industry organized an ASTM gasoline and diesel sulfur test method round robin (June 2001 ASTM meeting)**
- **ASTM round robin test precision results were presented at June 2002 ASTM meeting**



HIGHWAY DIESEL RULE

15 ppm

**Designated Test Method
ASTM D 6428-99**

**Allowable Alternative Test Methods
ASTM D 5453-99 and ASTM D 3120-96**

500 ppm

**Designated Test method
ASTM D 2622-98**

**Allowable Alternative Test methods
ASTM D 5453-99 and ASTM D 6428-99**



TWO TYPES OF TEST VARIABILITY

Repeatability (r) Conditions

- **Same Sample**
- **Same Laboratory**
- **Same Test Method**
- **Same Instrument**
- **Same Operator**

Reproducibility (R) Conditions

- **Same Sample**
- **Same Test Method**
- **Different Laboratories**
- **Different Instruments**
- **Different Operator**



ASTM DIESEL SULFUR 2002 ROUND ROBIN PROGRAM

Labs

39 Oil Industry Labs
13 Instrument Vendor Labs
4 Independent Labs

Test Methods

ASTM D 6428	16 Labs
ASTM D 5453	40 Labs
ASTM D 3120	8 Labs
ASTM D 2622	28 Labs

Test Fuels

16 Gasolines (LT 1 to 400 ppm Sulfur)
16 Diesels (LT 1 to 35 ppm sulfur)

Overall

A Total of 92 Lab Units
A Total of 5,632 Data Points



ASTM DIESEL SULFUR 2002 ROUND ROBIN RESULTS

Diesel @ 15 ppm

Repeatability (r) Reproducibility (R)

ASTM D 6428

Global

3.06

15.55

APS

0.39

11.41

Antek

2.98

13.96

ASTM D 5453

1.20

6.26

ASTM D 3120

1.33

8.07



INDUSTRY CONCERNS

- Pipelines set their point of origin specifications based on the compliance standard and reproducibility (R).

ULSD Compliance = 15 ± 16 ppm (using ASTM D 6428)

- Based on these results, refiners will need to blend to impossibly low levels of sulfur on each batch at large cost.
- High potential for increased reprocessing of batches and/or downgrading of batches into other products both at the refinery and downstream.
- If the designated test method lacks good precision, use of an allowed (and more precise) alternative test method does not help the refiner or other parties, including EPA enforcement.



NEXT ASTM STEPS

- **ASTM will update the four sulfur test methods (6428, 5453, 3120, 2622) with the recent ASTM round-robin precision results.**
- **The ASTM D 6428 test method will be balloted within ASTM as a diesel sulfur test method.**



INDUSTRY RECOMMENDATION

Recommendation

EPA should choose the most precise test method as the designated method for measuring ULSD sulfur, while allowing alternative test methods to qualify

