

Challenges and prospects for certification and quality control of aviation biofuels

Fábio da Silva Vinhado
Coordinator of CPT

**Making Sustainable Alternative Fuels Viable
in Brazil**

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anp
Agência Nacional
do Petróleo,
Gás Natural e Biocombustíveis

- Research and Technological Analysis Center (CPT) is the laboratory of ANP (Brasília).
- Actually CPT/ANP is a set of laboratories focused on Fossil Fuels, Biofuels, Lubricants and Crude Oil.



- Modern facilities: completed renovated in 2015;
- ISO 17025 accredited laboratory;
- ASTM D02 Member.
- WGs Coordination under ISO/TC 28, helping to develop standards for fuel ethanol and biodiesel quality control.
- Central laboratory of Fuel Quality Monitoring System in Brazil
- Proficiency testing provider for fuels and lubricants.

- Developing activities (essays and reports) on Jet Fuel: ANP, CENIPA, SERIPA IV and Federal Police;
- Infrastructure to meet approximately 60% of ASTM D7566





Designation: D7566 – 18

An American National Standard

Standard Specification for Aviation Turbine Fuel Containing Synthesized Hydrocarbons¹

Alternative Jet Fuels pathways - approved

Id	Feedstock	Products	% with Jet	Status	Producers
FT-SPK	Coal, Natural Gas or Biomass	Iso & N-Paraffins	50	ASTM D7566 Annex A1	SASOL, Syntroleum, Shell
HEFA	Oils and Fats	Iso & N-Paraffins	50	ASTM D7566 Annex A2	UOP, Syntroleum, Neste,
SIP	Sugar	Paraffin	10	ASTM D7566 Annex A3	Amyris
FT-SPK/A	Coal, Natural Gas or Biomass	Iso & N-Paraffins and Aromatics	50	ASTM D7566 Annex A4	SASOL, Syntroleum, Shell
ATJ	Ethanol and Butanol	Iso & N-Paraffins	50	ASTM D7566 Annex A5	GEVO, Cobalt, Lanzatech

Alternative Jet Fuels pathways - under tests

Id	Feedstock	Products	Status	Producers
CHJ	Oils and Fats	Iso, N- and Cyclo-paraffins and Aromatics	Tier 3 and 4 of ASTM D4054	ARA
SAK	Sugars	Iso & N-Paraffins and Aromatics	Tier 1 and 2 concl. Evaluat. by OEMs	Virent
IH ²	Celulosic residues	Iso, N- and Cyclo-paraffins and Aromatics	Tier 1 and 2	Shell
HFP-HEFA	Oils and Fats	Iso & N-Paraffins rich in N-Paraf	Tier 1 and 2 concl. Evaluat. by OEMs	Green Diesel
ATJ-SKA	Ethanol and Butanol	Iso & N-Paraffins and Aromatics	Tier 1 and 2	Biogy, Swed Biofuels
IHI Bb Oil	Microalgae	Iso & N-Paraffins	Tier 1 and 2	IHI

D1655 (RANP 37/2009)	Table 1 D7566 (RANP 63/2014)	Table A2.1 D7566
<p>COMPOSITION</p> <p>Acidity, total mgKOH/g</p> <p>1.Aromatics, percent by volume</p> <p>1.Aromatics, percent by volume</p> <p>Sulfur, mercaptan, percent by mass</p> <p>Sulfur, total percent by mass</p>	<p>COMPOSITION</p> <p>Acidity, total mgKOH/g</p> <p>1.Aromatics, percent by volume</p> <p>1.Aromatics, percent by volume</p> <p>Sulfur, mercaptan, percent by mass</p> <p>Sulfur, total percent by mass</p>	<p>COMPOSITION</p> <p>Acidity, total mgKOH/g</p>
<p>VOLATILITY</p> <p>Distillation temperature, °C</p> <p>10 % recovered, temperature</p> <p>50 % recovered, temperature</p> <p>90 % recovered, temperature</p> <p>Final boiling point, temperature</p> <p>Distillation residue, %</p> <p>Distillation loss, %</p> <p>Flash point, °C</p> <p>Density at 15 °C, kg/m³</p>	<p>VOLATILITY</p> <p>Distillation temperature, °C</p> <p>10 % recovered, temperature</p> <p>50 % recovered, temperature</p> <p>90 % recovered, temperature</p> <p>Final boiling point, temperature</p> <p>Distillation residue, %</p> <p>Distillation loss, %</p> <p>Flash point, °C</p> <p>Density at 15 °C, kg/m³</p>	<p>VOLATILITY</p> <p>Both of the following requirements shall be met:</p> <p>Physical distillation</p> <p>Distillation temperature, °C</p> <p>10 % recovered, temperature (T10)</p> <p>50 % recovered, temperature (T50)</p> <p>90 % recovered, temperature (T90)</p> <p>Final boiling point, temperature</p> <p>T90 – T10</p> <p>Distillation residue, %</p> <p>Distillation loss, %</p> <p>Simulated distillation</p> <p>Distillation temperature, °C</p> <p>10 % recovered, temperature (T10)</p> <p>50 % recovered, temperature (T50)</p> <p>90 % recovered, temperature (T90)</p> <p>Final boiling point, temperature</p>
<p>FLUIDITY</p> <p>Freezing point, °C</p> <p>Viscosity – 20 °C, mm²/s</p>	<p>FLUIDITY</p> <p>Freezing point, °C</p> <p>Viscosity – 20 °C, mm²/s</p>	<p>Flash point, °C</p> <p>Density at 15 °C, kg/m³</p> <p>Freezing point, °C</p>

D1655 (RANP 37/2009) cont.	Table 1 D7566 (RANP 63/2014) cont.	Table A2.1 D7566 cont.
<p>COMBUSTION Net heat of combustion, MJ/kg One of the following requirements shall be met: (1) Smoke point, mm, or (2) Smoke point, mm, and Naphthalenes, vol, %</p>	<p>COMBUSTION Net heat of combustion, MJ/kg One of the following requirements shall be met: (1) Smoke point, mm, or (2) Smoke point, mm, and Naphthalenes, vol, %</p>	<p>Existent gum, mg/100 mL FAME, ppm</p>
<p>CORROSION Copper strip, 2 h at 100 °C</p>	<p>CORROSION Copper strip, 2 h at 100 °C</p>	
<p>THERMAL STABILITY (2.5 h at control temperature of 260 °C min) Filter pressure drop, mm Hg Tube rating: One of the following requirements shall be met (1)Annex A1 VTR, VTR Color Code (2)Annex A2 ITR or Annex A3 ETR, mm average over area of 2.5 mm²</p>	<p>THERMAL STABILITY (2.5 h at control temperature of 260 °C min) Filter pressure drop, mm Hg Tube rating: One of the following requirements shall be met (1)Annex A1 VTR, VTR Color Code (2)Annex A2 ITR or Annex A3 ETR, mm average over area of 2.5 mm²</p>	<p>Thermal stability (2.5 h at control temperature) Temperature, °C Filter pressure drop, mm Hg Tube rating: One of the following requirements shall be met (1)Annex A1 VTR, VTR Color Code (2)Annex A2 ITR or Annex A3 ETR, mm average over area of 2.5 mm²</p>
<p>CONTAMINANTS Existent gum, mg/100 mL Microseparator, Rating Without electrical conductivity additive With electrical conductivity additive ADDITIVES Electrical conductivity, pS/m</p>	<p>CONTAMINANTS Existent gum, mg/100 mL Microseparator, Rating Without electrical conductivity additive With electrical conductivity additive ADDITIVES Electrical conductivity, pS/m</p>	<p>ADDITIVES Antioxidants, mg/L</p>

D1655 (RANP 37/2009) cont.	Table 1 D7566 (RANP 63/2014) cont.	Table A2.1 D7566 cont.
	<p>Part 2 - Extended requirements</p> <p>COMPOSITION</p> <p>One of the following requirements shall be met:</p> <p>1.Aromatics, percent by volume</p> <p>1.Aromatics, percent by volume</p> <p>VOLATILITY</p> <p>Distillation</p> <p>T50 – T10, °C</p> <p>T90 – T10, °C</p> <p>LUBRICITY</p> <p>Lubricity, mm</p> <p>FLUIDITY</p> <p>Viscosity – 40 °C, mm²/s</p>	<p>Table A2.2 D7566</p> <p>Hydrocarbon Composition</p> <p>Cycloparaffins, mass percent</p> <p>Aromatics, mass percent</p> <p>Paraffins, mass percent</p> <p>Carbon and hydrogen, mass percent</p> <p>Non-Hydrocarbon Composition</p> <p>Nitrogen, mg/kg</p> <p>Water, mg/kg</p> <p>Sulfur, mg/kg</p> <p>Metals (Al, Ca, Co, Cr, Cu, Fe, K, Mg, etc), mg/kg</p> <p>Halogens, mg/kg</p>

❖ **Common tests in fuel laboratories:**

Acidity, Distillation, Flash Point, Density, Gum, Water and Sulfur

❖ **Also can be found:**

FAME (IP 585), SimDis and CHN

❖ **Specific Tests:**

Freezing point, JFTOT, Lubricity, Cycloparaffins, Aromatics, Paraffins, Nitrogen and Halogens

Specific Test Method	Estimated price of the instrument (R\$)
Freezing point	40.000,00
JFTOT	700.000,00
Lubricity BOCLE	500.000,00
Nitrogen Analyzer (D4629)	450.000,00
Combustion ion Chromatography (D7359)	750.000,00
TOTAL	2.400.000,00

About

❖ **Thermal Stability (JFTOT)**

Failure reported with D3241.

Instruments not equivalent (differences observed in tubes)

Need to improve metrology requirements

❖ **Composition Hydrocarbons (ASTM D2425, GCXGC)**

D2425 is the method on the spec

Complex method that has previously shown some limitations in differentiating between isoparaffins and cycloparaffins (and does not differentiate between n- and isoparaffins)

GCXGC is used to determine composition, but is not standardized (no precision data)

- ❖ **Method to detect content of biojet (ASTM D6866)**

Maybe for the future

- ❖ **Development of reference materials**

Essential to ensure the quality control.

- ❖ **Development of field tests (for both renewable and fossil fuel)**

Resolution ANP 37/2009 (spec for Jet A1) and Resolution ANP 63/2014 (aviation turbine synthesized hydrocarbons) under revision:

The main aim is to update according to ASTM D1655 and D7566

-Updating test methods and annexes A4 (SPK/A) and A5 (ATJ) of D7566

Publication scheduled for the next semester of 2018

- ❖ Initial investments in laboratories to have the complete infrastructure for the certification of renewable aviation fuels

- ❖ Laboratories with expertise in aviation fuels

- ❖ Tools to ensure the proficiency of the laboratories:
 - Interlaboratory programs
 - Developing of reference materials

- ❖ Test Methods Development aligned with ASTM

E-mail: fvinhado@anp.gov.br

☎ +55 (61) 3426 5181

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