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R&D partner in chemistry

# Characterization of regenerated methyl methacrylate (rMMA) and derived polymer (rPMMA)


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# Characterization

- Methyl methacrylate monomer (virgin & regenerated)
  - Purity and composition
  - Odour concentration

- Poly(methyl methacrylate) (based on virgin or recycled monomer)
  - Global odour intensity and odorous notes
  - Odour concentration
  - Nature (virgin vs recycled monomer)

# Techniques and methodologies

| Technique | Principle | Results/deliverable |
|---|---|---|
| **High Performance Liquid Chromatography (HPLC)** | Separation by liquid chromatography, identification by ultra-violet, refractive index | • Quantification of target compounds |
| **Field of odours® methodology (FO)** | Human panel of trained experts | • Global odour intensity<br>• decomposition into fundamental notes and their intensities |
| **Gas chromatography-mass spectrometry/field ionization detector (GC-MS/FID)** | Separation by gas chromatography, identification by MS, quantification by FID | • Chemical composition<br>• Quantification of target compounds |
| **Dynamic olfactometry according to EN13725 (DO)** | • Human panel representative of the whole population<br>• Determination of dilution to reach 50 % detection by panel | • Detection threshold<br>• Odour quantification in $uo_E/m^3$ |

# Techniques and methodologies

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## High Performance Liquid Chromatography (HPLC)

## Gas chromatography-mass spectrometry/field ionization detector (GC-MS/FID)

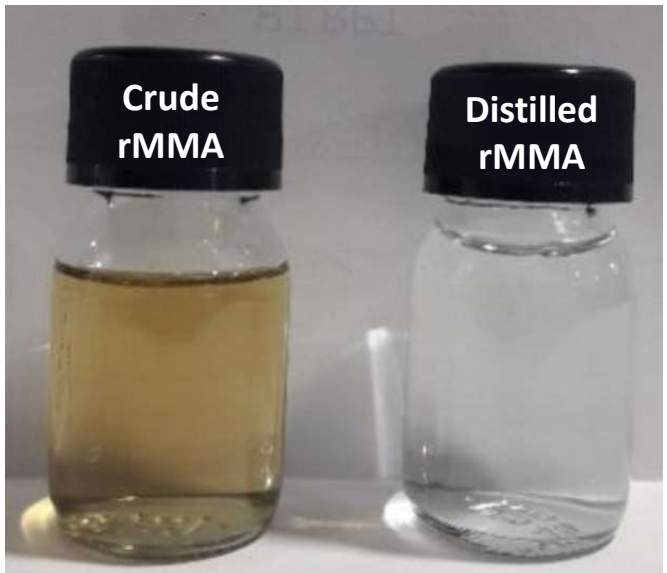
## Field of odours® methodology (FO)

## Dynamic olfactometry according to EN13725 (DO)

# Practical examples (1)

## Purity and composition determination of distilled rMMA by GC-MS/FID (liquid injection):

| | Virgin MMA | rMMA 1 | rMMA 2 | rMMA 3 | rMMA 4 |
|---|---|---|---|---|---|
| **MMA purity** | **99,96** | **99,80** | **99,88** | **99,68** | **99,84** |
| Methyl Acrylate (MeA) * | ND | ND | ND | ND | ND |
| Ethyl Acrylate (EA) * | ND | 0,12 | 0,05 | 0,04 | ND |
| Methyl isobutyrate** | ND | 0,02 | / | 0,02 | 0,02 |
| Others | 0,04 | 0,06 | 0,07 | 0,27 | 0,14 |

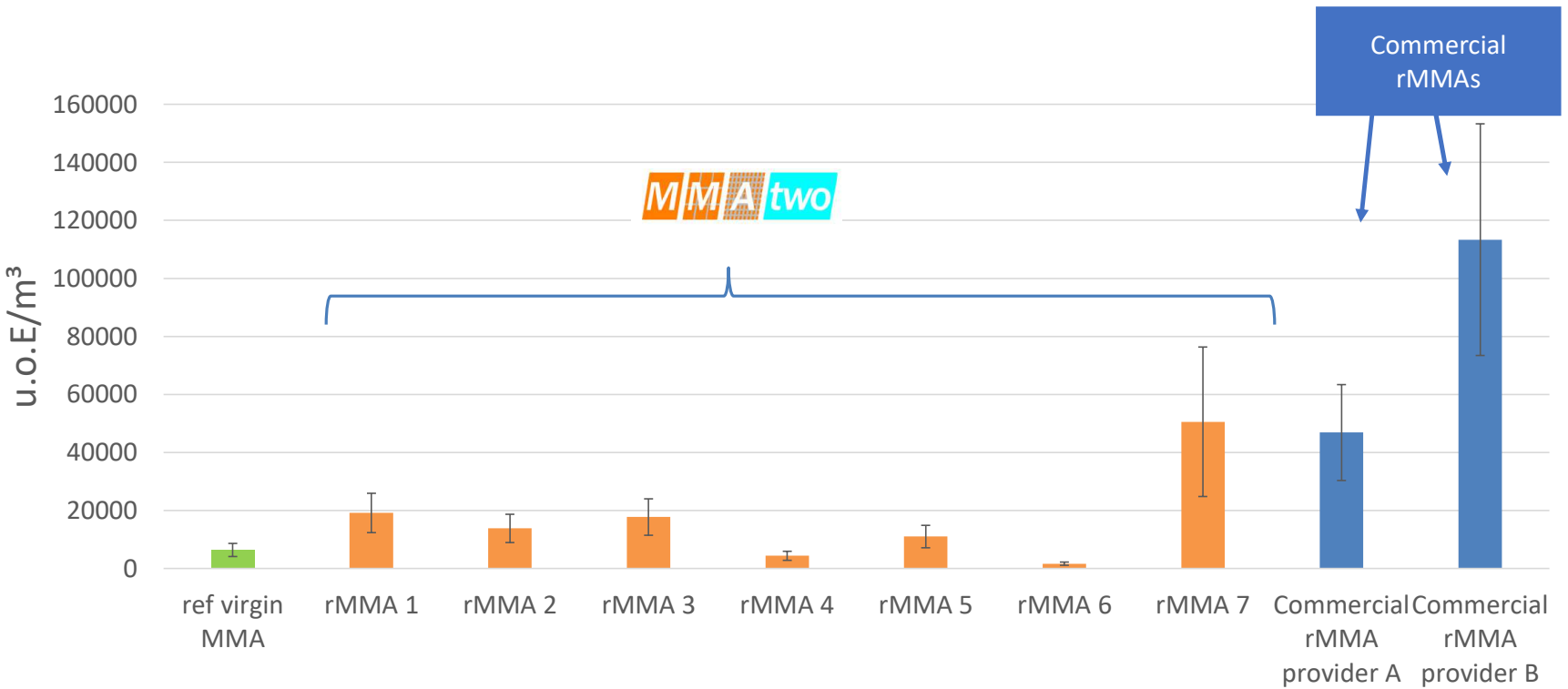



* Comonomers used in extr/inj PMMA grades
** Non-intentionally added substance (NIAS), representative of PMMA depolymerisation
ND : Not detected

(All values in % of total FID area)

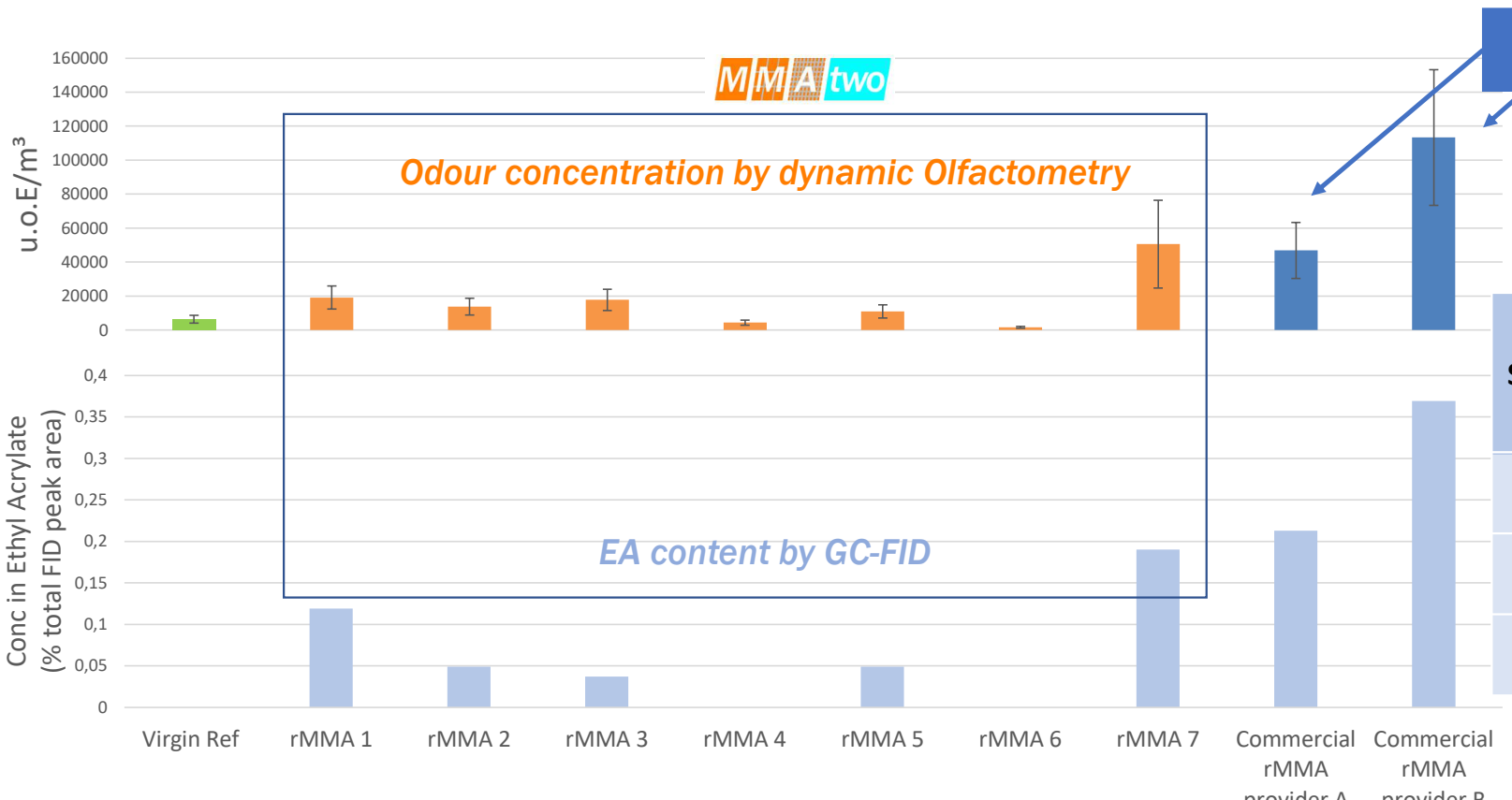
# Practical examples (2)

**Odour concentration of various rMMAs vs virgin MMA by dynamic olfactometry allows ranking of products, including commercial benchmarks :**



# Practical examples (3)

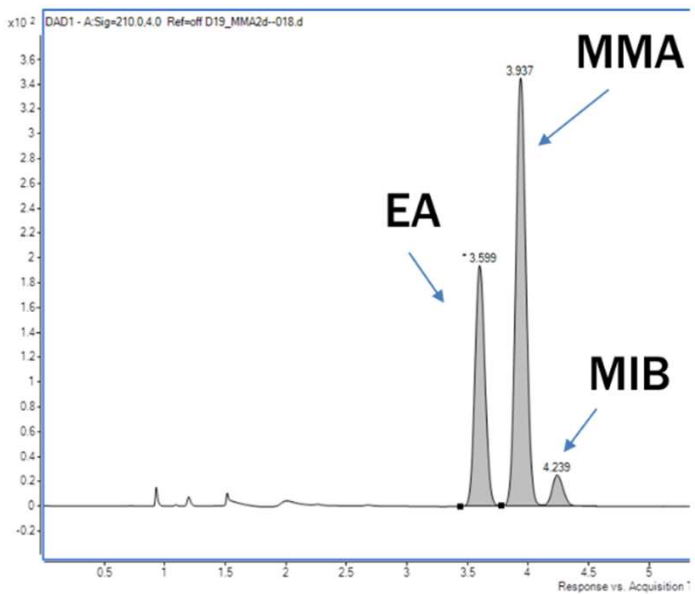
**Correlation between odour concentration and composition of various rMMAs evidences ethyl acrylate as main contributor to odour, as supported by odour detection thresholds:**




| Substance | Odour detection threshold (µg/m³) |
|---|---|
| MMA | 22,03 |
| MIB | 16,10 |
| EA | **0,08** |

# Practical examples (4)

**Successful Identification of nature of PMMA waste stream by high performance liquid chromatography:**



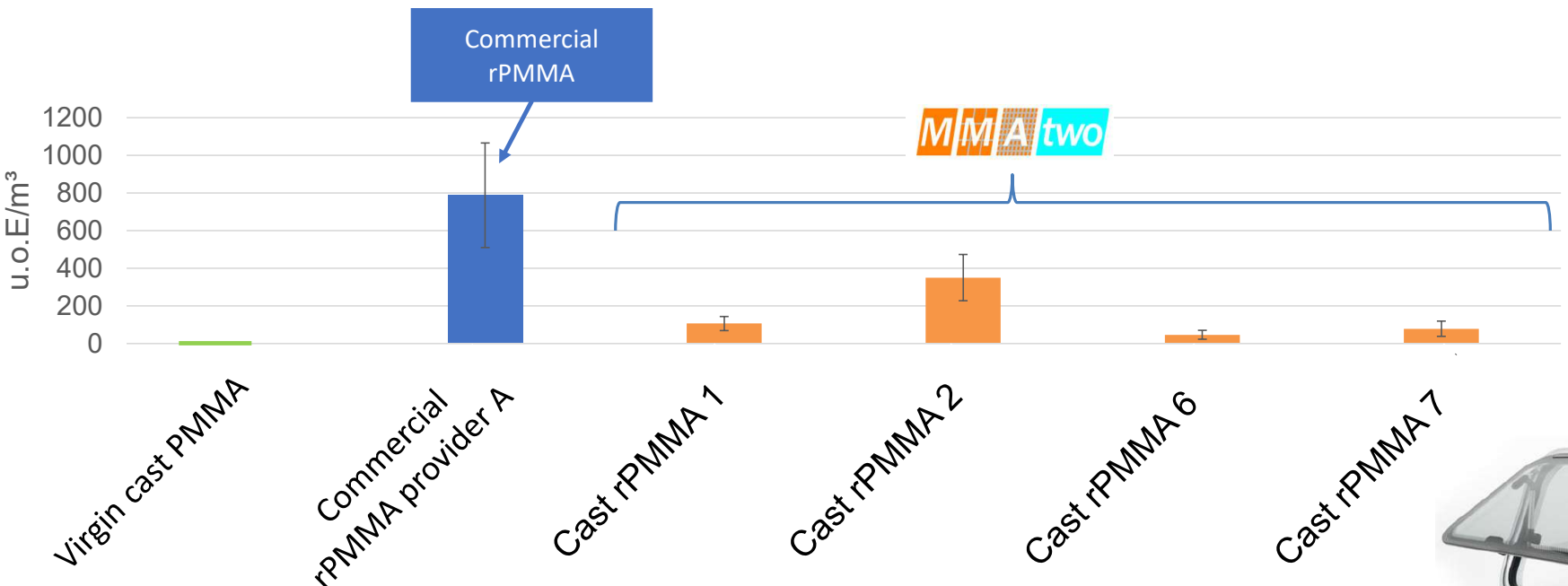

HPLC chromatograms of standards
(10 minutes analysis time)

**Main results**

→ Identification of MMA, ethyl acrylate (EA) and methyl isobutyrate (MIB)
**EA = tracer for ext/inj PMMA grade**
**MIB = tracer for recycled PMMA**

# Practical examples (5)

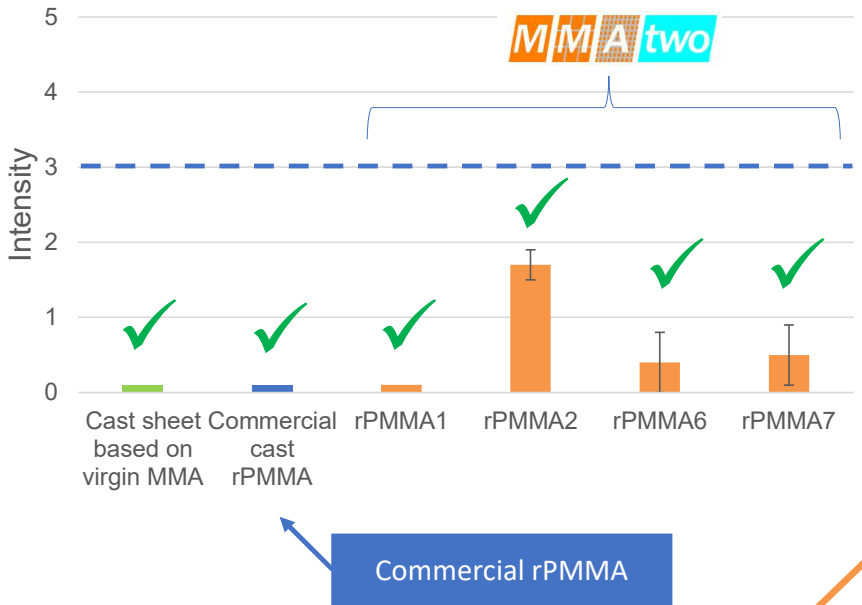
**Odour ranking of cast PMMA sheets for optical applications (caravan windows) based on rMMA, using dynamic olfactometry:**




*Picture: courtesy Delta Glass BV*

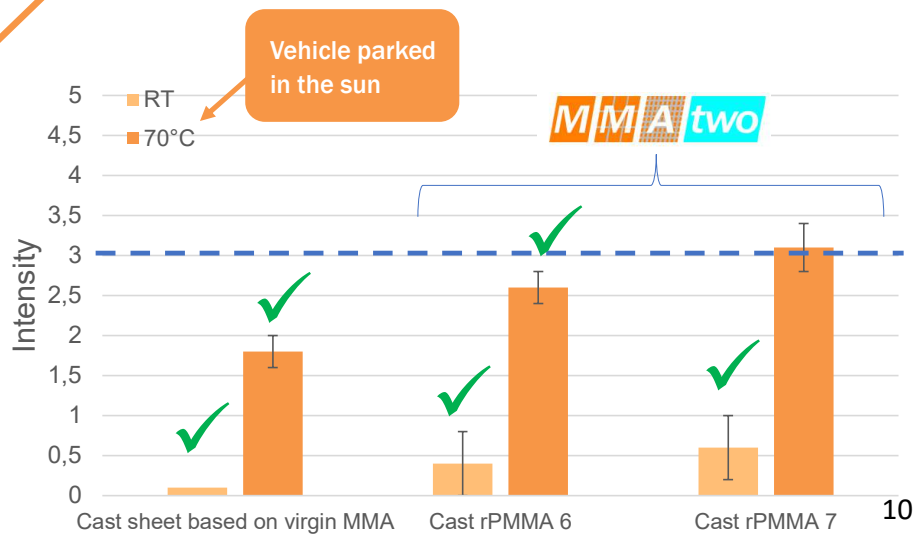
# Practical examples (6)

## Odour evaluation of cast PMMA sheets for optical applications (caravan windows) based on rMMA, using global odour intensity at RT and 70°C :



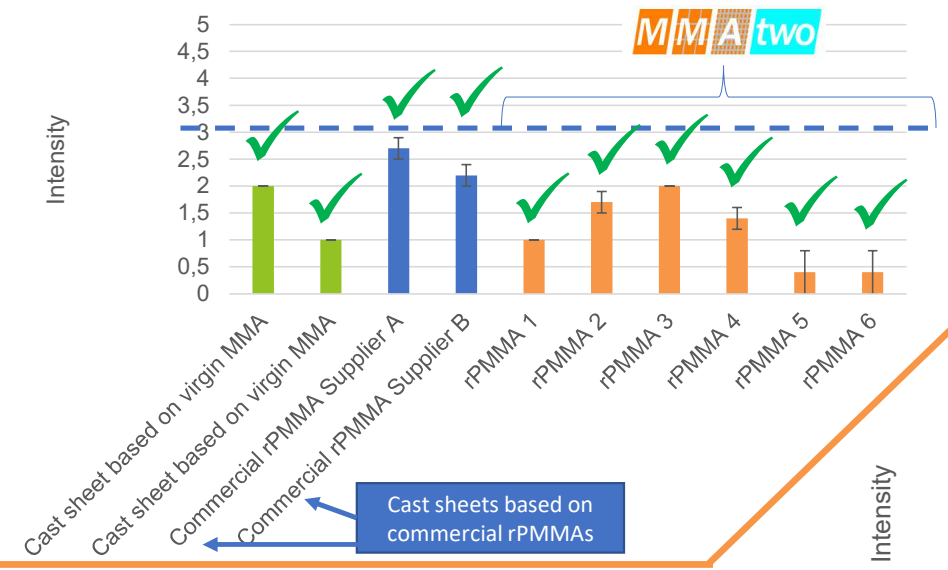

- All samples < 3 = automotive interior specification
- rPMMA 2 : sligthly more odorous
- Other samples = virgin MMA

- At 30 °C : virgin PMMA < rPMMA << 3
- At 70 °C : virgin PMMA < rPMMA ≤ 3
- Main notes : Acrid

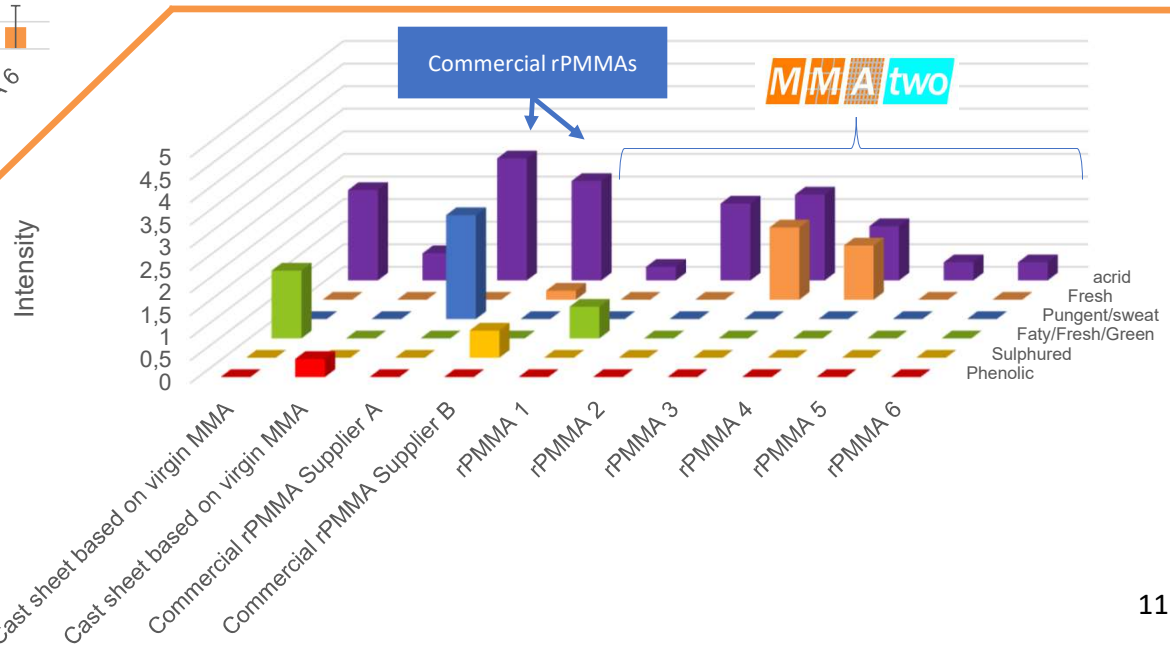


# Practical examples (7)

**Odour evaluation of cast PMMA sheets for composite applications based on rMMA, using global odour intensity and Field of odours®:**




- All samples < 3 = automotive interior specification
- MMAtwo rPMMA = Virgin PMMA < rPMMAs based on commercial rPMMAs from suppliers A and B




- Acrid intensity ≈ Global intensity
- Pungent note detected in rPMMA based on commercial rMMA from supplier A
- A few fresh and fatty notes detected in MMAtwo samples

# Conclusions

Certech can deliver characterization services on regenerated or virgin methyl methacrylate and derived polymers :

- Odour global intensity
- Odourous notes : types and intensity
- Odour concentration
- Composition and purity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