



## Premium Certified Reference Material RRM Fe-112

**PRODUCT: IRON ORE**  
**Pilbara Region Western Australian**  
**CERTIFICATE OF ANALYSIS**

Certificate Number: RRM CRM Fe-112 Rev 000

Date: 3 June 2017

Table 1 Fe-112 Constituents (ISO 2596:2006 Hygroscopic Moisture Corrected)

| Analyte (Unit)                     | Assigned value | Standard Deviation |                    | 95% Confidence Limits |       | Number of Laboratories | Number of Analyses | Mean of number of analyses per Laboratory |
|------------------------------------|----------------|--------------------|--------------------|-----------------------|-------|------------------------|--------------------|---|
|                                    |                | Within Laboratory  | Between Laboratory | Lower                 | Upper |                        |                    |   |
| Fe (%)                             | 57,83          | 0,18               | 0,20               | 57,75                 | 57,91 | 12                     | 36                 | 3,0                                       |
| Fe calculated (%)                  | 57,86          | 0,06               | 0,08               | 57,82                 | 57,89 | 12                     | 36                 | 3,0                                       |
| SiO <sub>2</sub> (%)               | 4,67           | 0,05               | 0,05               | 4,65                  | 4,69  | 12                     | 36                 | 3,0                                       |
| Al <sub>2</sub> O <sub>3</sub> (%) | 2,96           | 0,02               | 0,04               | 2,94                  | 2,98  | 12                     | 36                 | 3,0                                       |
| TiO <sub>2</sub> (%)               | 0,099          | 0,003              | 0,004              | 0,098                 | 0,101 | 12                     | 36                 | 3,0                                       |
| Mn (%)                             | 0,228          | 0,001              | 0,002              | 0,227                 | 0,230 | 12                     | 36                 | 3,0                                       |
| CaO (%)                            | 0,047          | 0,003              | 0,006              | 0,044                 | 0,050 | 12                     | 36                 | 3,0                                       |
| MgO (%)                            | 0,077          | 0,007              | 0,020              | 0,065                 | 0,088 | 12                     | 36                 | 3,0                                       |
| K <sub>2</sub> O (%)               | 0,010          | 0,001              | 0,002              | 0,009                 | 0,011 | 12                     | 36                 | 3,0                                       |
| Na <sub>2</sub> O (%)              | 0,016          | 0,005              | 0,011              | 0,009                 | 0,024 | 7                      | 19                 | 2,7                                       |
| P (%)                              | 0,043          | -                  | 0,001              | 0,042                 | 0,043 | 12                     | 36                 | 3,0                                       |
| S (%)                              | 0,034          | 0,001              | 0,002              | 0,033                 | 0,035 | 11                     | 32                 | 2,9                                       |
| LOI-371 %                          | 7,73           | 0,04               | 0,09               | 7,64                  | 7,82  | 3                      | 27                 | 9,0                                       |
| LOI-425 %                          | 8,02           | 0,03               | 0,06               | 7,99                  | 8,05  | 9                      | 44                 | 4,9                                       |
| LOI-650 %                          | 8,58           | 0,04               | 0,07               | 8,54                  | 8,61  | 9                      | 44                 | 4,9                                       |
| LOI-1000 %                         | 8,83           | 0,06               | 0,09               | 8,80                  | 8,87  | 15                     | 63                 | 4,2                                       |
| Cl (%)                             | 0,004          | -                  | 0,002              | 0,002                 | 0,005 | 7                      | 19                 | 2,7                                       |
| As (%)                             | 0,008          | 0,001              | 0,001              | 0,007                 | 0,008 | 7                      | 19                 | 2,7                                       |
| Ba (%)                             | -              | -                  | -                  | -                     | -     | 7                      | 19                 | 2,7                                       |
| Co (%)                             | 0,003          | 0,001              | 0,002              | 0,001                 | 0,004 | 7                      | 19                 | 2,7                                       |
| Cu (%)                             | 0,001          | -                  | 0,001              | -                     | 0,002 | 7                      | 19                 | 2,7                                       |
| Cr (%)                             | 0,005          | 0,001              | 0,001              | 0,004                 | 0,005 | 3                      | 6                  | 2,0                                       |
| Ni (%)                             | 0,001          | 0,001              | 0,002              | -                     | 0,003 | 6                      | 15                 | 2,5                                       |
| Pb (%)                             | 0,011          | 0,001              | 0,001              | 0,011                 | 0,012 | 7                      | 19                 | 2,7                                       |
| Sn (%)                             | 0,001          | -                  | 0,002              | -                     | 0,002 | 7                      | 19                 | 2,7                                       |
| Sr (%)                             | -              | -                  | -                  | -                     | -     | 6                      | 15                 | 2,5                                       |
| V (%)                              | -              | -                  | -                  | -                     | -     | 6                      | 15                 | 2,5                                       |
| Zn (%)                             | 0,001          | 0,001              | 0,001              | 0,001                 | 0,002 | 7                      | 19                 | 2,7                                       |
| Zr (%)                             | 0,004          | 0,001              | 0,002              | 0,003                 | 0,006 | 7                      | 19                 | 2,7                                       |

**Table 2 Fe-112 Constituents (non-hygroscopically moisture corrected)**

| Analyte (Unit)                     | Assigned value | Standard Deviation |                    | 95% Confidence Limits |       | Number of Laboratories | Number of Analyses | Mean of number of analyses per Laboratory |
|------------------------------------|----------------|--------------------|--------------------|-----------------------|-------|------------------------|--------------------|---|
|                                    |                | Within Laboratory  | Between Laboratory | Lower                 | Upper |                        |                    |   |
| Fe (%)                             | 57,64          | 0,10               | 0,16               | 57,56                 | 57,72 | 13                     | 50                 | 3,8                                       |
| Fe calculated (%)                  | 57,60          | 0,06               | 0,15               | 57,51                 | 57,68 | 13                     | 50                 | 3,8                                       |
| SiO <sub>2</sub> (%)               | 4,63           | 0,02               | 0,04               | 4,61                  | 4,66  | 13                     | 50                 | 3,8                                       |
| Al <sub>2</sub> O <sub>3</sub> (%) | 2,93           | 0,02               | 0,05               | 2,90                  | 2,96  | 13                     | 50                 | 3,8                                       |
| TiO <sub>2</sub> (%)               | 0,099          | 0,003              | 0,004              | 0,097                 | 0,100 | 13                     | 50                 | 3,8                                       |
| Mn (%)                             | 0,229          | 0,001              | 0,004              | 0,227                 | 0,232 | 10                     | 42                 | 4,2                                       |
| CaO (%)                            | 0,050          | 0,002              | 0,006              | 0,047                 | 0,053 | 13                     | 50                 | 3,8                                       |
| MgO (%)                            | 0,094          | 0,006              | 0,011              | 0,088                 | 0,099 | 13                     | 50                 | 3,8                                       |
| K <sub>2</sub> O (%)               | 0,012          | 0,001              | 0,002              | 0,011                 | 0,013 | 11                     | 44                 | 4,0                                       |
| Na <sub>2</sub> O (%)              | 0,023          | 0,005              | 0,006              | 0,021                 | 0,026 | 12                     | 47                 | 3,9                                       |
| P (%)                              | 0,043          | 0,001              | 0,001              | 0,042                 | 0,043 | 13                     | 50                 | 3,8                                       |
| S (%)                              | 0,034          | 0,001              | 0,002              | 0,033                 | 0,035 | 12                     | 46                 | 3,8                                       |
| LOI-371 %                          | 7,97           | 0,05               | 0,11               | 7,92                  | 8,03  | 11                     | 55                 | 5,0                                       |
| LOI-425 %                          | 8,26           | 0,06               | 0,07               | 8,23                  | 8,30  | 7                      | 35                 | 5,0                                       |
| LOI-650 %                          | 8,81           | 0,05               | 0,11               | 8,76                  | 8,87  | 12                     | 56                 | 4,7                                       |
| LOI-1000 %                         | 9,18           | 0,12               | 0,28               | 9,05                  | 9,30  | 16                     | 77                 | 4,8                                       |
| Cl (%)                             | 0,002          | -                  | 0,001              | 0,001                 | 0,003 | 8                      | 30                 | 3,8                                       |
| As (%)                             | 0,010          | 0,001              | 0,002              | 0,008                 | 0,011 | 9                      | 33                 | 3,7                                       |
| Ba (%)                             | -              | -                  | -                  | -                     | -     | 5                      | 19                 | 3,8                                       |
| Co (%)                             | 0,002          | -                  | 0,002              | 0,001                 | 0,003 | 10                     | 38                 | 3,8                                       |
| Cu (%)                             | 0,004          | 0,001              | 0,002              | 0,003                 | 0,006 | 10                     | 35                 | 3,5                                       |
| Cr (%)                             | 0,006          | -                  | 0,001              | 0,006                 | 0,006 | 8                      | 27                 | 3,4                                       |
| Ni (%)                             | 0,003          | 0,001              | 0,001              | 0,003                 | 0,004 | 3                      | 9                  | 3,0                                       |
| Pb (%)                             | 0,012          | 0,001              | 0,001              | 0,011                 | 0,012 | 5                      | 17                 | 3,4                                       |
| Sn (%)                             | 0,002          | 0,001              | 0,002              | -                     | 0,003 | 5                      | 17                 | 3,4                                       |
| Sr (%)                             | 0,001          | -                  | 0,003              | -                     | 0,005 | 3                      | 8                  | 2,7                                       |
| V (%)                              | -              | -                  | -                  | -                     | -     | 2                      | 7                  | 3,5                                       |
| Zn (%)                             | 0,002          | 0,001              | 0,001              | 0,001                 | 0,003 | 4                      | 11                 | 2,8                                       |
| Zr (%)                             | 0,008          | 0,001              | 0,002              | 0,007                 | 0,010 | 4                      | 13                 | 3,3                                       |

### Introduction:

Certified Reference Materials (CRMs) are used by laboratories to prove the value of their service offerings and for clients of laboratories to evaluate and monitor laboratory performance. CRMs must comply with high metrological requirements and ensuring traceability of measurement results.

Since most techniques employing analytical instrumentation are comparative, these techniques require a sample of known composition (CRM) for accurate calibration. Grade and Matrix matched CRM's are thus vital to the core of the analytical chemistry industry.

## **Preparation of Material:**

The material constituting RRM Fe-112 has been according to ISO 17034:2016, and includes the following:

- Drying to constant mass
- Crushing and dry milling to nominal 53µm particle size
- Homogenization
- Systematic rotary division of the entire lot, to final aliquot
- Packaging
- Rotary divided to nominal 250g sealed jars
- Rotary divided to nominal 10g geochem pouches, vacuum sealed in barrier foil
- Custom package sizes available on request

Unique to RRM, rotary division of all material to final packaging.

## **Methods of Analysis:**

The analysis of the test samples has been conducted according to each individual laboratory's routine analytical procedures. The material has been dried at 105°C until constant mass was achieved, or corrected for moisture according to ISO 2596:2006. Each test sample has been prepared, analyzed and reported in duplicate; with results reported on a dry basis.

Elements and Oxides determined as follows:

- Multi-element Iron Ore Suite – XRF fused disc analysis
- Loss on Ignition – Thermo Gravimetric Analysis

## **Homogeneity Evaluation:**

For the evaluation of the homogeneity of the Fe-112 material, 14 samples were selected throughout the batch for analysis in duplicate. ANOVA (one-way analysis of variance) was used to assess the homogeneity for measured Iron and total Loss on Ignition. No significant variation was observed for both 'batch' and 'within unit' exercises. The results and statistical evaluation from the interlaboratory studies (presented in Tables 3, 4, 5 and 6) further validate the homogeneity. The material can thus be considered fit for purpose for use as a CRM.

## **Statistical Evaluation:**

We have grouped the laboratories according to the method of determining moisture. Certain laboratories determine the dry weight of iron ore samples at 105°C, whilst some use the ISO 2596:2006 correction for hygroscopic moisture. Some iron ore types report different moisture values for each of these methods, which in turn influences the chemical analysis as the determined moisture content has an influence on the dry starting mass.

A comprehensive statistical evaluation of the results received from the various laboratories was performed. Outliers were identified and removed from the data sets for the certification exercise.

The assigned values are the mean of means after removal of outliers. All constituents reported with a between laboratory % RSD of <5% are regarded as certified values.

**Table 3 Fe-112 Statistical Evaluation (Hygroscopic Moisture Corrected)**

| Analyte (Unit)  | Fe (%) | Fe calc (%) | SiO <sub>2</sub> (%) | Al <sub>2</sub> O <sub>3</sub> (%) | TiO <sub>2</sub> (%) | Mn (%) | CaO (%) |
|---|--------|-------------|----------------------|------------------------------------|----------------------|--------|---------|
| <b>Assigned value</b>                                 | 57.83  | 57.86       | 4.67                 | 2.96                               | 0.099                | 0.228  | 0.047   |
| <b>Number of Laboratories</b>                         | 12     | 12          | 12                   | 12                                 | 12                   | 12     | 12      |
| <b>Number of Analyses</b>                             | 36     | 36          | 36                   | 36                                 | 36                   | 36     | 36      |
| <b>Between Laboratory Standard Deviation</b>          | 0.20   | 0.08        | 0.05                 | 0.04                               | 0.004                | 0.002  | 0.006   |
| <b>Within Laboratory Standard Deviation</b>           | 0.18   | 0.06        | 0.05                 | 0.02                               | 0.003                | 0.001  | 0.003   |
| <b>Between Laboratory Relative Standard Deviation</b> | 0.34 % | 0.13 %      | 1.14 %               | 1.22 %                             | 3.87 %               | 1.00 % | 12.0 %  |
| <b>Within Laboratory Relative Standard Deviation</b>  | 0.31 % | 0.13 %      | 1.09 %               | 0.72 %                             | 2.63 %               | 0.59 % | 5.61 %  |
| <b>Standard Uncertainty</b>                           | 0.040  | 0.10 %      | 0.010                | 0.009                              | 0.001                | 0.001  | 0.002   |
| <b>Lower confidence limit</b>                         | 57.75  | 57.82       | 4.65                 | 2.94                               | 0.098                | 0.227  | 0.044   |
| <b>Upper confidence limit</b>                         | 57.91  | 57.89       | 4.69                 | 2.98                               | 0.101                | 0.230  | 0.050   |
| <b>Lower limit of tolerance</b>                       | 57.44  | 57.70       | 4.56                 | 2.89                               | 0.092                | 0.224  | 0.036   |
| <b>Upper limit of tolerance</b>                       | 58.22  | 58.01       | 4.78                 | 3.03                               | 0.107                | 0.233  | 0.058   |

| Analyte (Unit)  | P (%)  | S (%)  | MgO (%) | K <sub>2</sub> O (%) | Na <sub>2</sub> O (%) | As (%) | Cl (%) |
|---|--------|--------|---------|----------------------|-----------------------|--------|--------|
| <b>Assigned value</b>                                 | 0.043  | 0.034  | 0.077   | 0.010                | 0.016                 | 0.004  | 0.008  |
| <b>Number of Laboratories</b>                         | 12     | 11     | 12      | 12                   | 7                     | 7      | 7      |
| <b>Number of Analyses</b>                             | 36     | 32     | 36      | 36                   | 19                    | 19     | 19     |
| <b>Between Laboratory Standard Deviation</b>          | 0.001  | 0.002  | 0.020   | 0.002                | 0.011                 | 0.002  | 0.001  |
| <b>Within Laboratory Standard Deviation</b>           | -      | 0.001  | 0.007   | 0.001                | 0.005                 | -      | 0.001  |
| <b>Between Laboratory Relative Standard Deviation</b> | 2.37 % | 5.29 % | 26 %    | 20 %                 | 64 %                  | 61 %   | 13.7 % |
| <b>Within Laboratory Relative Standard Deviation</b>  | 1.12 % | 2.21 % | 9.73 %  | 11.6 %               | 28 %                  | 10.4 % | 13.6 % |
| <b>Standard Uncertainty</b>                           | -      | 0.001  | 0.006   | 0.001                | 0.004                 | 0.001  | -      |
| <b>Lower confidence limit</b>                         | 0.042  | 0.033  | 0.065   | 0.009                | 0.009                 | 0.002  | 0.007  |
| <b>Upper confidence limit</b>                         | 0.043  | 0.035  | 0.088   | 0.011                | 0.024                 | 0.005  | 0.008  |
| <b>Lower limit of tolerance</b>                       | 0.041  | 0.031  | 0.036   | 0.006                | -                     | -      | 0.006  |
| <b>Upper limit of tolerance</b>                       | 0.045  | 0.038  | 0.117   | 0.014                | 0.038                 | 0.008  | 0.010  |

| Analyte (Unit)  | Co (%) | Cu (%) | Pb (%) | Zn (%) | Ba (%) | Cr (%) | Ni (%) |
|---|--------|--------|--------|--------|--------|--------|--------|
| <b>Assigned value</b>                                 | -      | 0.003  | 0.001  | 0.005  | 0.001  | 0.011  | 0.001  |
| <b>Number of Laboratories</b>                         | 7      | 7      | 7      | 3      | 6      | 7      | 7      |
| <b>Number of Analyses</b>                             | 19     | 19     | 19     | 6      | 15     | 19     | 19     |
| <b>Between Laboratory Standard Deviation</b>          | -      | 0.002  | 0.001  | 0.001  | 0.002  | 0.001  | 0.002  |
| <b>Within Laboratory Standard Deviation</b>           | -      | 0.001  | -      | 0.001  | 0.001  | 0.001  | -      |
| <b>Between Laboratory Relative Standard Deviation</b> | -      | 67 %   | 98 %   | 18.9 % | 134 %  | 5.60 % | 118 %  |
| <b>Within Laboratory Relative Standard Deviation</b>  | -      | 31 %   | 31 %   | 18.9 % | 94 %   | 5.60 % | 15.4 % |
| <b>Standard Uncertainty</b>                           | -      | 0.001  | -      | -      | 0.001  | -      | 0.001  |
| <b>Lower confidence limit</b>                         | -      | 0.001  | -      | 0.004  | -      | 0.011  | -      |

|                                 |   |       |       |       |       |       |       |
|---------------------------------|---|-------|-------|-------|-------|-------|-------|
| <b>Upper confidence limit</b>   | - | 0.004 | 0.002 | 0.005 | 0.003 | 0.012 | 0.002 |
| <b>Lower limit of tolerance</b> | - | -     | -     | 0.003 | -     | 0.010 | -     |
| <b>Upper limit of tolerance</b> | - | 0.006 | 0.004 | 0.006 | 0.005 | 0.012 | 0.004 |

| Analyte (Unit)  | Sn (%) | Sr (%) | V (%) | Zr (%) |
|---|--------|--------|-------|--------|
| <b>Assigned value</b>                                 | -      | -      | 0.001 | 0.004  |
| <b>Number of Laboratories</b>                         | 6      | 6      | 7     | 7      |
| <b>Number of Analyses</b>                             | 15     | 15     | 19    | 19     |
| <b>Between Laboratory Standard Deviation</b>          | -      | -      | 0.001 | 0.002  |
| <b>Within Laboratory Standard Deviation</b>           | -      | -      | 0.001 | 0.001  |
| <b>Between Laboratory Relative Standard Deviation</b> | -      | -      | 72 %  | 43 %   |
| <b>Within Laboratory Relative Standard Deviation</b>  | -      | -      | 45 %  | 30 %   |
| <b>Standard Uncertainty</b>                           | -      | -      | -     | 0.001  |
| <b>Lower confidence limit</b>                         | -      | -      | 0.001 | 0.003  |
| <b>Upper confidence limit</b>                         | -      | -      | 0.002 | 0.006  |
| <b>Lower limit of tolerance</b>                       | -      | -      | -     | 0.001  |
| <b>Upper limit of tolerance</b>                       | -      | -      | 0.003 | 0.008  |

Table 4 Fe-112 Statistical Evaluation (non-hygroscopically moisture corrected)

| Analyte (Unit)  | Fe (%) | Fe calc (%) | SiO <sub>2</sub> (%) | Al <sub>2</sub> O <sub>3</sub> (%) | TiO <sub>2</sub> (%) | Mn (%) | CaO (%) |
|---|--------|-------------|----------------------|------------------------------------|----------------------|--------|---------|
| <b>Assigned value</b>                                 | 57.64  | 57.60       | 4.63                 | 2.93                               | 0.099                | 0.229  | 0.050   |
| <b>Number of Laboratories</b>                         | 13     | 13          | 13                   | 13                                 | 13                   | 10     | 13      |
| <b>Number of Analyses</b>                             | 50     | 50          | 50                   | 50                                 | 50                   | 42     | 50      |
| <b>Between Laboratory Standard Deviation</b>          | 0.16   | 0.15        | 0.04                 | 0.05                               | 0.004                | 0.004  | 0.006   |
| <b>Within Laboratory Standard Deviation</b>           | 0.10   | 0.06        | 0.02                 | 0.02                               | 0.003                | 0.001  | 0.002   |
| <b>Between Laboratory Relative Standard Deviation</b> | 0.28 % | 0.27 %      | 0.92 %               | 1.83 %                             | 3.58 %               | 1.88 % | 11.1 %  |
| <b>Within Laboratory Relative Standard Deviation</b>  | 0.17 % | 0.10 %      | 0.50 %               | 0.57 %                             | 2.59 %               | 0.36 % | 3.64 %  |
| <b>Standard Uncertainty</b>                           | 0.040  | 0.042       | 0.011                | 0.015                              | 0.001                | 0.001  | 0.002   |
| <b>Lower confidence limit</b>                         | 57.56  | 57.51       | 4.61                 | 2.90                               | 0.097                | 0.227  | 0.047   |
| <b>Upper confidence limit</b>                         | 57.72  | 57.68       | 4.66                 | 2.96                               | 0.100                | 0.232  | 0.053   |
| <b>Lower limit of tolerance</b>                       | 57.31  | 57.29       | 4.55                 | 2.83                               | 0.092                | 0.221  | 0.039   |
| <b>Upper limit of tolerance</b>                       | 57.96  | 57.91       | 4.72                 | 3.04                               | 0.106                | 0.238  | 0.061   |

| Analyte (Unit)                               | P (%) | S (%) | MgO (%) | K <sub>2</sub> O (%) | Na <sub>2</sub> O (%) | As (%) | Cl (%) |
|--|-------|-------|---------|----------------------|-----------------------|--------|--------|
| <b>Assigned value</b>                        | 0.043 | 0.034 | 0.094   | 0.012                | 0.023                 | 0.002  | 0.010  |
| <b>Number of Laboratories</b>                | 13    | 12    | 13      | 11                   | 12                    | 8      | 9      |
| <b>Number of Analyses</b>                    | 50    | 46    | 50      | 44                   | 47                    | 30     | 33     |
| <b>Between Laboratory Standard Deviation</b> | 0.001 | 0.002 | 0.011   | 0.002                | 0.006                 | 0.001  | 0.002  |

|   |        |        |        |        |       |       |        |
|---|--------|--------|--------|--------|-------|-------|--------|
| <b>Within Laboratory Standard Deviation</b>           | 0.001  | 0.001  | 0.006  | 0.001  | 0.005 | -     | 0.001  |
| <b>Between Laboratory Relative Standard Deviation</b> | 3.46 % | 5.60 % | 11.2 % | 16.8 % | 26 %  | 69 %  | 25 %   |
| <b>Within Laboratory Relative Standard Deviation</b>  | 1.59 % | 2.68 % | 6.65 % | 11.6 % | 23 %  | 23 %  | 9.78 % |
| <b>Standard Uncertainty</b>                           | -      | 0.001  | 0.003  | 0.001  | 0.001 | -     | 0.001  |
| <b>Lower confidence limit</b>                         | 0.042  | 0.033  | 0.088  | 0.011  | 0.021 | 0.001 | 0.008  |
| <b>Upper confidence limit</b>                         | 0.043  | 0.035  | 0.099  | 0.013  | 0.026 | 0.003 | 0.011  |
| <b>Lower limit of tolerance</b>                       | 0.040  | 0.030  | 0.073  | 0.008  | 0.011 | -     | 0.005  |
| <b>Upper limit of tolerance</b>                       | 0.046  | 0.038  | 0.115  | 0.016  | 0.035 | 0.005 | 0.014  |

| Analyte (Unit)  | Co<br>(%) | Cu<br>(%) | Pb<br>(%) | Zn<br>(%) | Ba<br>(%) | Cr<br>(%) | Ni<br>(%) |
|---|-----------|-----------|-----------|-----------|-----------|-----------|-----------|
| <b>Assigned value</b>                                 | -         | 0.002     | 0.004     | 0.006     | 0.003     | 0.012     | 0.002     |
| <b>Number of Laboratories</b>                         | 5         | 10        | 10        | 8         | 3         | 5         | 5         |
| <b>Number of Analyses</b>                             | 19        | 38        | 35        | 27        | 9         | 17        | 17        |
| <b>Between Laboratory Standard Deviation</b>          | -         | 0.002     | 0.002     | 0.001     | 0.001     | 0.001     | 0.002     |
| <b>Within Laboratory Standard Deviation</b>           | -         | -         | 0.001     | -         | 0.001     | 0.001     | 0.001     |
| <b>Between Laboratory Relative Standard Deviation</b> | -         | 91 %      | 58 %      | 9.54 %    | 37 %      | 7.64 %    | 85 %      |
| <b>Within Laboratory Relative Standard Deviation</b>  | -         | 17.0 %    | 29 %      | 5.95 %    | 37 %      | 5.53 %    | 30 %      |
| <b>Standard Uncertainty</b>                           | -         | 0.001     | 0.001     | -         | -         | -         | 0.001     |
| <b>Lower confidence limit</b>                         | -         | 0.001     | 0.003     | 0.006     | 0.003     | 0.011     | -         |
| <b>Upper confidence limit</b>                         | -         | 0.003     | 0.006     | 0.006     | 0.004     | 0.012     | 0.003     |
| <b>Lower limit of tolerance</b>                       | -         | -         | -         | 0.005     | 0.001     | 0.010     | -         |
| <b>Upper limit of tolerance</b>                       | -         | 0.006     | 0.009     | 0.007     | 0.006     | 0.013     | 0.005     |

| Analyte (Unit)  | Sn<br>(%) | Sr<br>(%) | V<br>(%) | Zr<br>(%) |
|---|-----------|-----------|----------|-----------|
| <b>Assigned value</b>                                 | 0.001     | -         | 0.002    | 0.008     |
| <b>Number of Laboratories</b>                         | 3         | 2         | 4        | 4         |
| <b>Number of Analyses</b>                             | 8         | 7         | 11       | 13        |
| <b>Between Laboratory Standard Deviation</b>          | 0.003     | -         | 0.001    | 0.002     |
| <b>Within Laboratory Standard Deviation</b>           | -         | -         | 0.001    | 0.001     |
| <b>Between Laboratory Relative Standard Deviation</b> | 246 %     | -         | 55 %     | 22 %      |
| <b>Within Laboratory Relative Standard Deviation</b>  | 30 %      | -         | 41 %     | 14.7 %    |
| <b>Standard Uncertainty</b>                           | 0.002     | -         | -        | 0.001     |
| <b>Lower confidence limit</b>                         | -         | -         | 0.001    | 0.007     |
| <b>Upper confidence limit</b>                         | 0.005     | -         | 0.003    | 0.010     |
| <b>Lower limit of tolerance</b>                       | -         | -         | -        | 0.005     |
| <b>Upper limit of tolerance</b>                       | 0.007     | -         | 0.004    | 0.012     |

### LOI Certified Values:

The LOI statistics from the inter-laboratory study are presented in the table below:

**Table 5 Fe-112 LOI Certified Values (Hygroscopic Moisture Corrected)**

| Determination (Unit)                           | LOI-371 % | LOI-425 % | LOI-650 % | LOI-1000 % |
|--|-----------|-----------|-----------|------------|
| Assigned value                                 | 7.73      | 8.02      | 8.58      | 8.83       |
| Number of Laboratories                         | 3         | 9         | 9         | 15         |
| Number of Analyses                             | 27        | 44        | 44        | 63         |
| Between Laboratory Standard Deviation          | 0.09      | 0.06      | 0.07      | 0.09       |
| Within Laboratory Standard Deviation           | 0.04      | 0.03      | 0.04      | 0.06       |
| Between Laboratory Relative Standard Deviation | 1.12 %    | 0.69 %    | 0.77 %    | 1.04 %     |
| Within Laboratory Relative Standard Deviation  | 0.49 %    | 0.33 %    | 0.43 %    | 0.68 %     |
| Standard Uncertainty                           | 0.045     | 0.017     | 0.019     | 0.019      |
| Lower confidence limit                         | 7.64      | 7.99      | 8.54      | 8.80       |
| Upper confidence limit                         | 7.82      | 8.05      | 8.61      | 8.87       |
| Lower limit of tolerance                       | 7.56      | 7.91      | 8.44      | 8.65       |
| Upper limit of tolerance                       | 7.91      | 8.13      | 8.71      | 9.02       |

**Table 6 Fe-112 LOI Certified Values (non-hygroscopically corrected moisture)**

| Determination (Unit)                           | LOI-371 % | LOI-425 % | LOI-650 % | LOI-1000 % |
|--|-----------|-----------|-----------|------------|
| Assigned value                                 | 7.97      | 8.26      | 8.81      | 9.18       |
| Number of Laboratories                         | 11        | 7         | 12        | 16         |
| Number of Analyses                             | 55        | 35        | 56        | 77         |
| Between Laboratory Standard Deviation          | 0.106     | 0.066     | 0.105     | 0.276      |
| Within Laboratory Standard Deviation           | 0.046     | 0.058     | 0.051     | 0.122      |
| Between Laboratory Relative Standard Deviation | 1.33 %    | 0.80 %    | 1.19 %    | 3.01 %     |
| Within Laboratory Relative Standard Deviation  | 0.58 %    | 0.70 %    | 0.58 %    | 1.33 %     |
| Standard Uncertainty                           | 0.029     | 0.016     | 0.027     | 0.063      |
| Lower confidence limit                         | 7.915     | 8.229     | 8.76      | 9.05       |
| Upper confidence limit                         | 8.033     | 8.295     | 8.87      | 9.30       |
| Lower limit of tolerance                       | 7.762     | 8.129     | 8.60      | 8.62       |
| Upper limit of tolerance                       | 8.186     | 8.394     | 9.02      | 9.73       |

### Origin of Material:

This material originates from the iron ore deposits of the Hamersley Group of the Hamersley Iron Province, central Pilbara Craton in Western Australia. The Hamersley Group rocks are early Proterozoic sediments, i.e. shale, siltstone, mudstone, chert, carbonate rocks and banded iron formations. Some minor volcanic units are also present in the Hamersley Group stratigraphy. There are two banded iron ore deposits within the Hamersley Group, the Brockman Iron Formation (approximately  $2481 \pm 4$  Ma) and the older Marra Mamba Iron Formation (approximately  $2597 \pm 5$  Ma). The Brockman Formation consists of cherts, mudstone, siltstone and banded iron formations; and is the main iron ore zone within the Hamersley Group. The iron formation consists of significant amounts of secondary enriched martite-goethite, which has an iron content of between 56 and 63 wt.%. The Marra Mamba Iron Formation is a surface enriched banded iron formation consisting of martite microplaty hematite ores, containing 60 to 68 wt.% iron.

1. Killick, M. F., H. M. Churchward, R. R. Anand (2003) Hamersley Iron Province, Western Australia. CRC LEME, Exploration and Mining.
2. Morris, R. C., M. Kneeshaw, (2011): Genesis modelling for the Hamersley BIF-hosted iron ores of Western Australia: A critical review. Australian Journal of earth sciences 58.

### Minor Elements and Specific Gravity:

Informational values for minor elements are provided; results from Lazer Ablation ICP-MS and for Specific Gravity by Helium Pycnometer (results are for a single analysis at one laboratory):

**SG (Helium Pycnometer) 4.29**

**Table 7 Fe-112 Minor Elements**

|                |               |                |                |                |                |                |                |
|----------------|---------------|----------------|----------------|----------------|----------------|----------------|----------------|
| As ppm<br>21,2 | Ba ppm<br>36  | Be ppm<br>0,2  | Bi ppm<br>0,2  | Cd ppm<br>0,3  | Ce ppm<br>23,3 | Co ppm<br>7    | Cs ppm<br>0,1  |
| Cu ppm<br>34   | Dy ppm<br>1,3 | Er ppm<br>0,9  | Eu ppm<br>0,38 | Ga ppm<br>2,5  | Gd ppm<br>1,25 | Ge ppm<br>4,55 | Hf ppm<br>1,7  |
| Ho ppm<br>0,29 | In ppm<br>-   | La ppm<br>5,31 | Lu ppm<br>0,17 | Mn ppm<br>2380 | Mo ppm<br>1    | Nb ppm<br>1,77 | Nd ppm<br>5,95 |
| Ni ppm<br>72   | Pb ppm<br>29  | Pr ppm<br>1,61 | Rb ppm<br>0,55 | Re ppm<br>-    | Sb ppm<br>1,2  | Sc ppm<br>6,3  | Se ppm<br>-    |
| Sm ppm<br>1,27 | Sn ppm<br>1,4 | Sr ppm<br>4,3  | Ta ppm<br>0,66 | Tb ppm<br>0,21 | Te ppm<br>-0,2 | Th ppm<br>1,92 | Tl ppm<br>-    |
| Tm ppm<br>0,17 | U ppm<br>1,89 | V ppm<br>25,1  | W ppm<br>1,4   | Y ppm<br>9,14  | Yb ppm<br>0,99 | Zn ppm<br>60   | Zr ppm<br>61   |

### XRD Analysis:

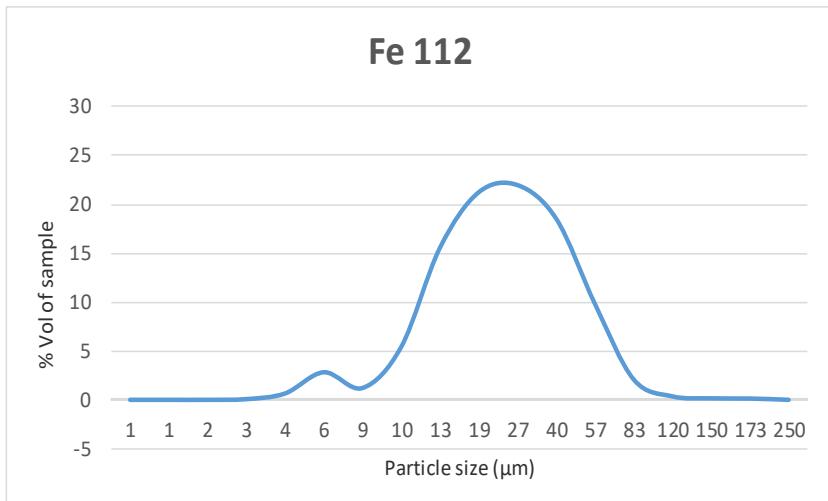
A sample of Fe-112 was submitted for Quantitative X-Ray Diffraction Analysis after drying at 50°C.  
 XRDQUANT01 - Quantitative analysis, crystalline and amorphous content

**Table 8 Fe-112 Mineral Abundance (XRD)**

| Mineral  | % mass fraction |
|--|-----------------|
| Hematite $\text{Fe}_2\text{O}_3$                       | 22              |
| Goethite $\text{FeO}(\text{OH})$                       | 59              |
| Magnetite $\text{Fe}_3\text{O}_4$                      | ND              |
| Kaolin $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ | ND              |
| Quartz $\text{SiO}_2$                                  | 2               |
| Amorphous content                                      | 17              |

### Particle Size Distribution:

A sample of Fe-112 was submitted for particle size analysis by MICROTRAC S3500 Lazer Diffraction System.



**Figure 1 Fe-112 Particle Size Distribution**

### Spectral Analysis:

A sample of Fe-112 was submitted for analysis by TerraSpec 4 VNIR-SWIR.

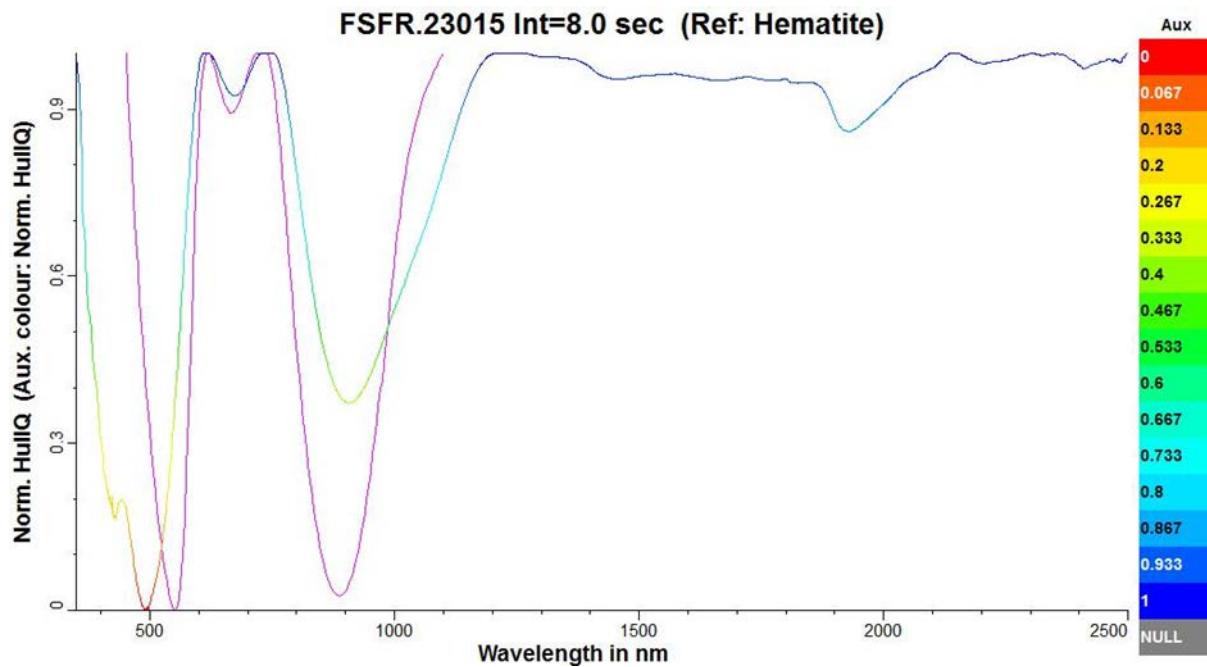


Figure 2 Fe-112 VNIR

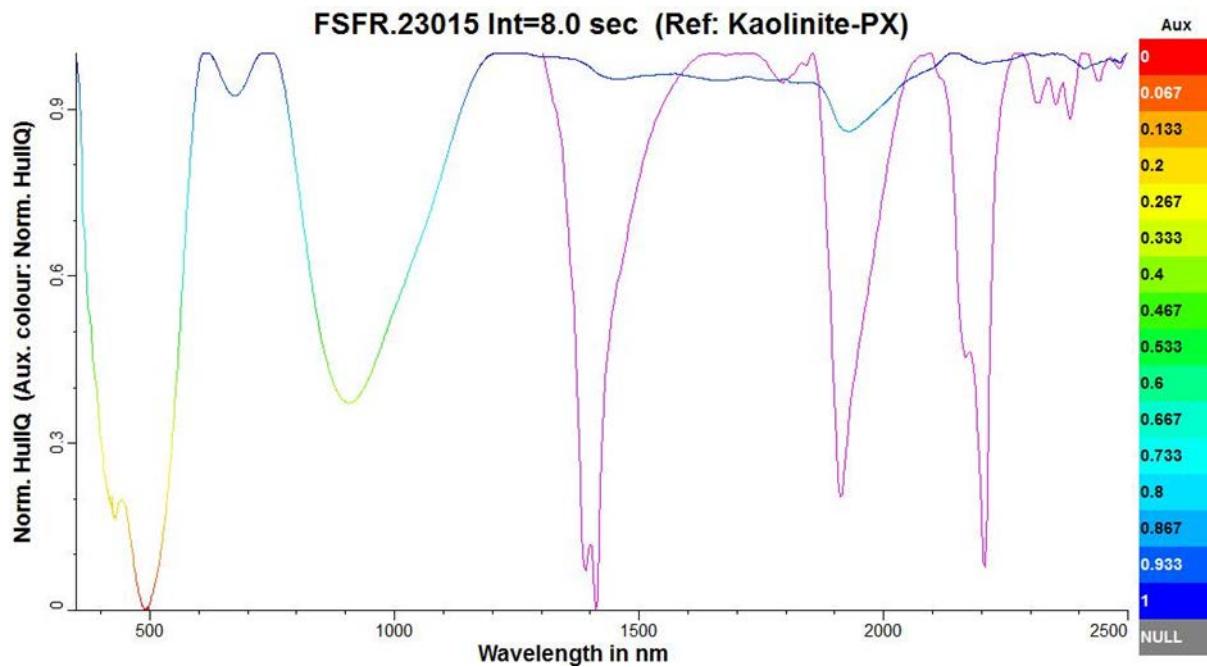
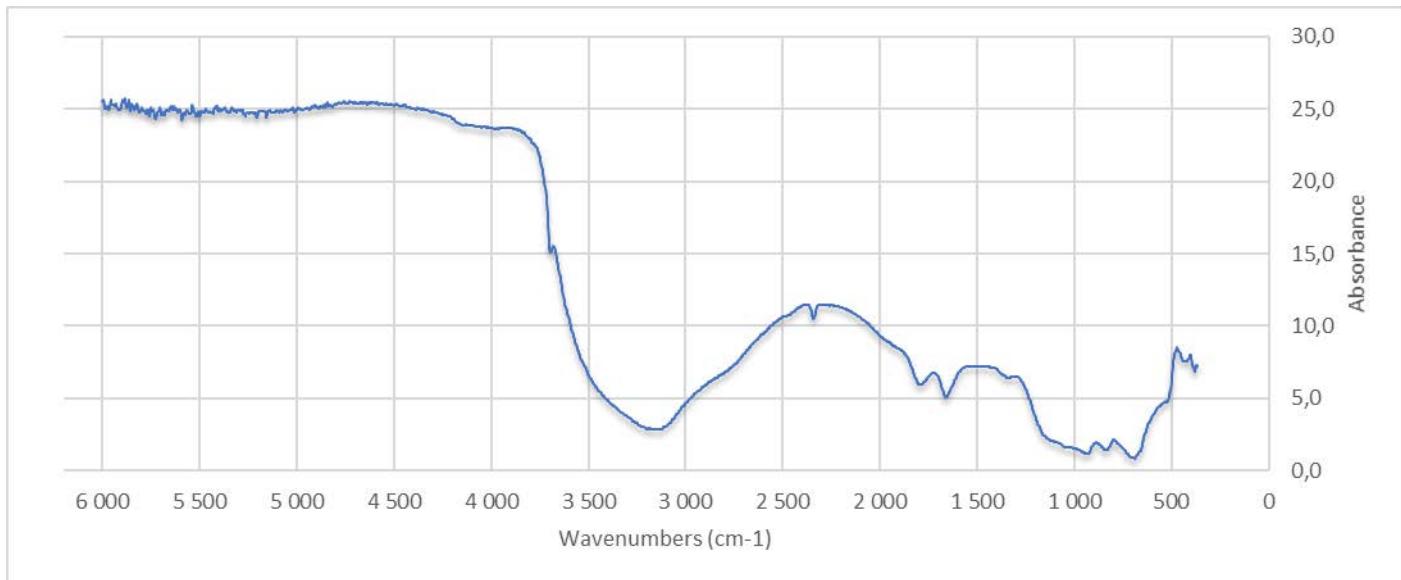


Figure 3 Fe-112 SWIR

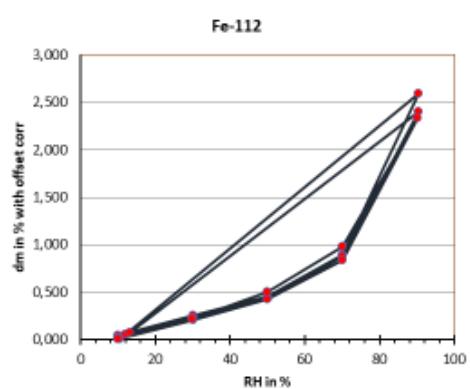
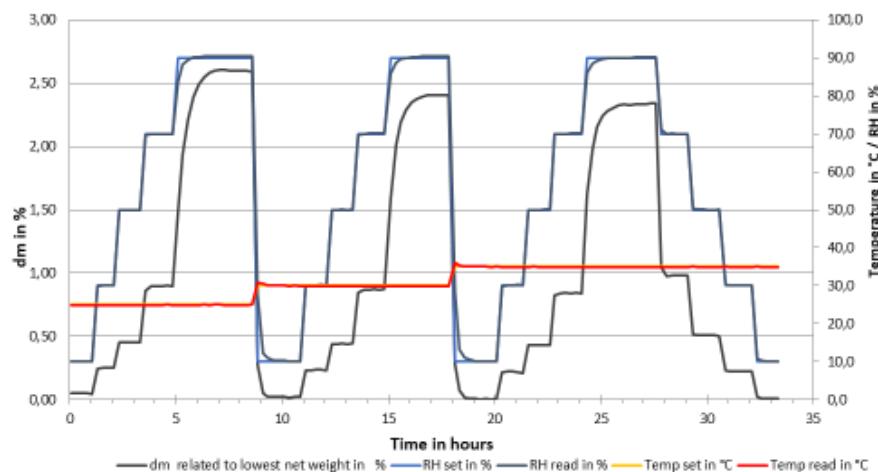
## FTIR Analysis:



**Figure 4** Fe-112 FTIR Spectra

## Hygroscopic Moisture:

To illustrate the effect of hygroscopic moisture a sample of RRM Fe-112 underwent water vapour sorption testing.



**Figure 5** Fe-112 Sorption Testing

From the tests it is clear that hygroscopic moisture could have an impact on the quality of the chemical analysis results and appropriate storage and pre-treatment is highly recommended.

**Participating Laboratories:**
**Table 9 Fe-112 Participating Laboratories**

|  |
|--|
| ALS Metallurgy, Australia                                |
| ALS Iron Ore, Australia                                  |
| BHP Billiton Nelson Point Laboratory, Australia          |
| BHP Billiton Newman Laboratory Line 1, Australia         |
| BHP Billiton Newman Laboratory Line 2, Australia         |
| BHP Billiton Newman Laboratory Line 3, Australia         |
| BHP Billiton Newman Laboratory Line 4, Australia         |
| Bureau Veritas, South Australia                          |
| Bureau Veritas, Western Australia                        |
| Citic Pacific Mining Laboratory, Australia               |
| IMP Automation, Australia                                |
| Intertek Maddington, Australia                           |
| Intertek Robotic Laboratories Anderson Point, Australia  |
| Intertek Robotic Laboratories Christmas Creek, Australia |
| Intertek Robotic Laboratories Cloudbreak, Australia      |
| Intertek Robotic Laboratories FMG Port, Australia        |
| Intertek Robotic Laboratories Mt Webber, Australia       |
| Intertek Robotic Laboratories Roy Hill Port, Australia   |
| Intertek Robotic Laboratories Solomon, Australia         |
| Intertek, South Africa                                   |
| Kumba Iron Ore Sishen Mine Laboratory, South Africa      |
| MinAnalytical Laboratory Services, Australia             |
| Rio Tinto Cape Lambert Port A Laboratory, Australia      |
| Rio Tinto Cape Lambert Port B Laboratory, Australia      |
| Rio Tinto Dampier Laboratory, Australia                  |
| Rio Tinto Marandoo Laboratory, Australia                 |
| Rio Tinto Paraburadoo Laboratory, Australia              |
| Rio Tinto West Angelas Laboratory, Australia             |
| Rio Tinto Yandicoogina Laboratory, Australia             |
| Set Point Laboratories, South Africa                     |

### **Intended Use:**

The Certified Reference Material RRM Fe-112 is intended to be used in analytical laboratories that analyse samples of similar grades (as presented in this certificate of analysis) and matrix.

- Monitoring of routine laboratory performance (both internal and external)
- Method development and method validation
- Instrument calibration

### **Instructions before Use:**

Safety precautions (in-line with safe laboratory practices) for handling fine particulate matter are advised; such as the use of safety glasses, dust masks, gloves and a laboratory coats.

### **Minimum Sample Size:**

- Multi-element Iron Ore Suite – XRF fused disc analysis 0.7g
- LOI – 1g

### **Analysis Validity:**

The property values for RRM Fe-112 Certified Reference Material remain valid provided that good laboratory practice is observed during handling and storage.

The material is in fine powder form and may be hygroscopic in nature.

### **Stability and storage:**

This CRM is considered to have long-term stability under normal storage conditions. The CRM is available in different packaging and mass lots. When considering the lot size and said the ISO 16042, which recommends that the total contents of the container holding the CRM prior to its first use be divided in two portions that match the intended test portions.

### **Traceability:**

The characterization of this material has been achieved by inter-laboratory study, each laboratory using an appropriate analytical method. These methods are calibrated against and analysed against high purity materials and appropriate Certified Reference Materials.

### **Additional Information:**

Additional information on material RRM Fe-112 is available on request and includes the following:

- Data Tables
- Statistical Tables
- Particle Size Analysis Data
- Quantitative X-Ray Diffraction Analysis Report
- FTIR Spectral Data
- VNIR-SWIR Spectral Data
- Sorption Testing Data

## Revisions:

This certificate is version 0.00 for the material RRM-Fe-112; and is intended to be a 'live document' intended to reflect progress in analytical chemistry. In that, any significant new data and information could be added at any time to ensure the currency and relevance of the certification. Any revisions to this Certificate of Analysis will be made available via the company website [www.resourcerefencematerials.com](http://www.resourcerefencematerials.com)

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## Prepared and Supplied:

Certified Reference Material RRM Fe-112 has been prepared, certified and is supplied by;

Resource Reference Materials (Pty) Ltd  
36 Michelson Road  
Anderbolt, Boksburg  
South Africa, 1459  
E-Mail: [info@resourcerefencematerials.com](mailto:info@resourcerefencematerials.com)

Approved on behalf of Resource Reference Materials (Pty) Ltd:



3<sup>rd</sup> June 2017

|          |                    |
|----------|--------------------|
| Name     | C.S. van der Linde |
| Position | Managing Director  |



## References:

- ISO 17034:2016** General requirements for the competence of reference material producers
- DRAFT ISO GUIDE 35:2016 Reference materials** — Guidance for the characterization and the assessment of the homogeneity and stability of the material
- ISO 5725-2:1994** Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method
- ISO 2596:2006 Iron ores** — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods
- ISO 11536:2015 Iron ores** — Determination of loss on ignition — Gravimetric method
- ISO 9516-1:2003 Iron ores** — Determination of various elements by X-ray fluorescence spectrometry — Part 1: Comprehensive procedure
- ISO 2597: 2006 part 3 Iron ores** — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods

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