**Phase Identification Exercise**

|  |  |
| --- | --- |
| **ID #** | **Phase** |
| 1 |  |
| 2 |  |
| 3 |  |
| 4 |  |
| 5 |  |
| 6 |  |
| 7 |  |
| 9 |  |
| 10 |  |
| 11 |  |
| 14 |  |
| 15 |  |
| 16 |  |
| 18 |  |
| 20 |  |
| 21 |  |
| 22 |  |
| 23 |  |
| 24 |  |
| 25 |  |
| 26 |  |

A set of individual phase diffraction patterns with d&I files are provided in the file Phase Identification Exercise.pdf. Using the key lines table provided here, identify the phase that produced each of the powder diffraction patterns and write your identifications in the table to the left.

You may wish to note some of the diagnostic peaks for each phase in the table as well.

Keep in mind that some deviations from peak position and relative intensity are normal, and since some of these patterns are from selective extractions, there may be a second (or maybe a third) phase present that could be identified after the primary phase has been identified.





**SRM Clinker Exercise**

The SRM clinkers were selected to reflect the range of textures and compositions of North American clinker production. The XRD data set consists of replicate determinations (with sample re-packing) of bulk clinker that has been ground to –10 μm and insoluble residues from the KOSH and SAM extractions. These extractions are useful for phase identification and examination for the presence of minor phases, which may be included in the final analysis of the bulk clinker.

The SAM extractions are typically performed quantitatively, allowing the XRD results to be recalculated on a whole-cement basis. The SAM insoluble residue mass fractions are provided below. The KOSH extraction is not quantitative and because some phases (periclase, for example) occur in both, an estimate as the difference between the SAM and unity are often biased. It is useful for examination of the silicate phases for identification.

Each clinker data set has a set of images of the diffraction patterns as well as text files listing peak d-spacing and relative intensities. Compare these to the cement phase d&I charts to establish your tentative phase identification.

Identify the component phases using the SAM and KOSH residues, compare these findings to the bulk clinker. You may make your identifications using the d&I lists and the diagnostic peaks or by reading the patterns into Profex and calling the stick figures from the Reference Structures. Recall that both approaches may not include the low intensity peaks. A rule of thumb in distinguishing a diffraction peak is that it must be roughly three standard deviations above background, essentially a small, but distinct peak. Finally, keep in mind that some secondary products may form. Portlandite is occasionally seen as a broad peak centered at 18 degrees two-theta in the SAM residue as a secondary product.

Record your phase identifications and the crystal structure lattice parameters for comparison to the final bulk clinker analysis, or for setting the initial lattice parameter values and restricting the bounds to facilitate the refinement.

Report the individual bulk phase estimates and compare them to the SRM certificate values and to the ASTM C1365 qualification limits.

The data sets consist of SRM 2686a, SRM2687, and SRM2688 and an extra data, 2687a, intended as a replacement for SRM2687

SAM Extraction Summary:

SRM2686a: 18.7%

SRM 2687: 17.2 %

SRM 2688: 17.2 %

Precision is an assessment of the variability one may expect when the test method is used by one or more reasonably competent laboratories and may be expressed as a standard deviation (1-σ). This is termed repeatability for the within-laboratory variability and reproducibility for the between-laboratory variability. ASTM repeatability (r) and reproducibility (R) are derived by multiplying the appropriate standard deviation by the factor 1.96\*√2, representing a 95% coverage factor

Compare your standard deviation for the three replicates against the d2s values given in Table 1 for the within-lab repeatability. Since we have reference materials, we may assess bias relative to the ILS participants by taking the difference between the SRM certificate value and the mean of the three replicates, using Table 2.

Table 1 Permissible Maximum Difference Between Replicate Values (percent of clinker or cement)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  | **Repeatability**  **Within-Lab** | | **Reproducibility**  **Between-Lab** | |
|  | **s-within** | **d2s-within** | **s-between** | **d2s-between** |
| Alite | 0.74 | 2.04 | 2.27 | 6.30 |
| Belite | 0.64 | 1.77 | 1.40 | 3.87 |
| Aluminate | 0.47 | 1.31 | 0.79 | 2.19 |
| Ferrite | 0.49 | 1.36 | 0.89 | 2.47 |
| Periclase | 0.23 | 0.63 | 0.50 | 1.39 |
| Arcanite | 0.22 | 0.60 | 0.34 | 0.94 |
| Gypsum | 0.21 | 0.59 | 0.59 | 1.65 |
| Bassanite | 0.39 | 1.08 | 0.58 | 1.60 |
| Anhydrite | 0.27 | 0.74 | 0.64 | 1.77 |
| Calcite\* | 0.21 | 0.58 | 1.00 | 2.78 |

\* proposed values from the Proficiency Test Program

Table 2 Permissible Maximum Difference Between Mean Value and Known Value (Mass Percent) Expressed at a 95 % Confidence Level for a Mean of a Selected Number of Replicates (k) = 2, 3, 4

|  |  |  |  |
| --- | --- | --- | --- |
| **Phase** | **2 replicates** | **3 Replicates** | **4 Replicates** |
| Alite | 5.93 | 4.91 | 4.31 |
| Belite | 3.70 | 3.06 | 2.69 |
| Aluminate | 2.14 | 1.77 | 1.55 |
| Ferrite | 2.46 | 2.04 | 1.79 |
| Periclase | 0.77 | 0.64 | 0.56 |
| Arcanite | 0.85 | 0.70 | 0.61 |
| Gypsum | 1.55 | 1.28 | 1.12 |
| Bassanite | 1.52 | 1.26 | 1.11 |
| Anhydrite | 1.67 | 1.38 | 1.21 |
| Calcite\* | 1.53 | 1.27 | 1.12 |

\* proposed values from the Proficiency Test Program

**Cement XRD Exercise**

Identify the phases using the KOSH and SAM residues, perform an analysis of the SAM residue (optional for the KOSH), re-calculate the SAM residue concentrations on a whole-cement basis. Refine the bulk cement diffraction data using the phases you identified in the first step, record the mass fractions and compare your results to those from the inter-laboratory consensus means provided in Table 3.

Since there are replicate cans for both the SAM and bulk analyses, a test result could be the average of one bulk and one recalculated SAM analyses or just a bulk cement analysis. Using the SAM residue provides a better view of the interstitial phases (remember you have eliminated about 75 % of the sample in performing a SAM extraction, so phases that may be obscure or not apparent in the bulk analysis are likely to be visible in the SAM extraction diffraction patterns. Report the data used for the test result.

SAM residue for CCRL 177 (A) is 23.7 % and for CCRL 178 (B) is 25.3 %.

Table x. Trimmed Mean by Phase for ASTM ILS Cements A and B

|  |  |  |
| --- | --- | --- |
| ASTM XRD ILS |  |  |
|  | ILS A (CCRL177) | ILS B (CCRL 178) |
| Alite | 60.0 | 63.4 |
| Belite | 16.7 | 12.3 |
| Aluminate | 4.2 | 3.6 |
| Ferrite | 9.5 | 9.9 |
| Periclase | 1.0 | 1.5 |
| Calcite | 2.5 | 2.5 |
| Arcanite | 0.7 |  |
| Gypsum |  | 5.0 |
| Bassanite | 1.9 |  |
| Anhydrite | 1.2 |  |