Appendix:

Tables of TOF-ND results.

The radiographic images were taken at the Center for X-ray tomography at the University of Ghent.

In the section “complementary information” results are reported as far as they contribute to the clarification of TOF-ND results. A full description of the PGAA data is given in [Kis et al. 2008].

“Box Alignment” in the set-up section refers to the positioning of the box in the sample chamber with respect to (wrt) the nominal diffractometer centre.

- “no offset”: centre of box coincides with centre of diffractometer;

In the section “Reality check” some details of the actual construction are given. A full description of the PGAA data is given in [Kirfel et al. 2008].

Table 1 (a). *TOF Neutron Diffraction results on D-II*

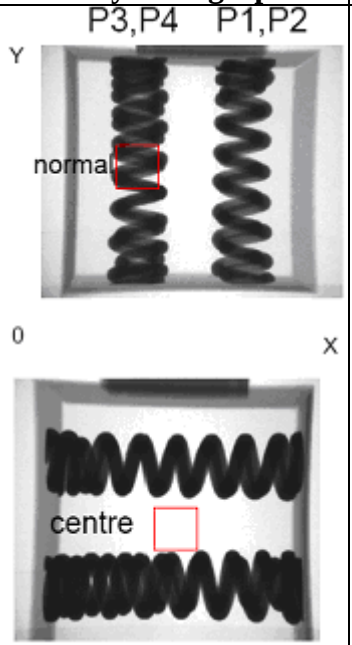
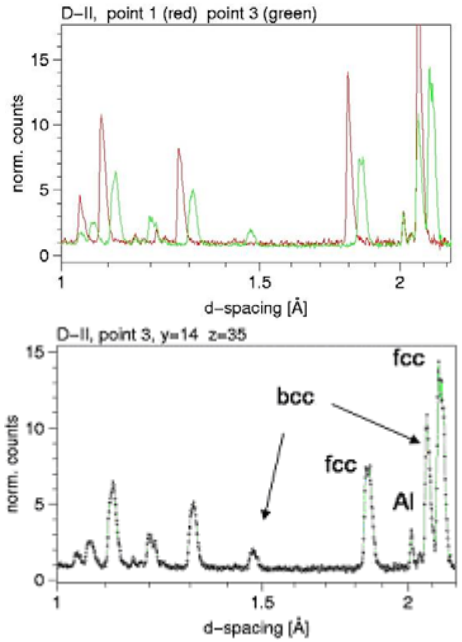
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
D-II		**Instrument: ROTAX** beam size 10x10 mm; beam along y Central box alignment: no offset; P1-P4: aligned with incident beam. “normal”: incident beam normal to the spiral. Beam traverses two spirals P3-P4. no offset “centre”: incident neutron beam aimed at box centre to determine filling. no offset		**Point 1**: fcc a=3.60 Å (Cu-type); **Point 2**: fcc a=3.601 Å (Cu-type) **Point 3**: fcc a=3.69 Å (Cu-type) (80wt%); bcc-phase (20wt%), a=2.94 Å **Point 4**: fcc a=3.686 Å (Cu-type) (85wt%), bcc-phase (15wt%), a=2.95 Å **Centre**: beam along z, empty (only Al peaks) **Normal** to the spiral, beam along z: fcc a=3.690 Å (Cu-type) (85wt%), bcc-phase (15wt%) a=2.94Å

Table 1 (b). *Comments on TOF-ND results on D-II*

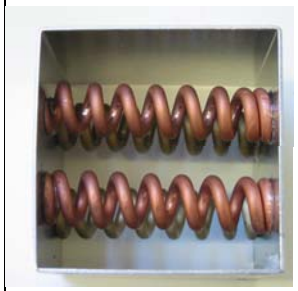
Box no.	Box content from TOF-ND	Complementary info	Reality Check
D-II	- Springs made of different materials but combination of two phases (fcc+bcc) - The lattice parameter indicates a **copper alloy** (bronze/brass) for the fcc metal, and beta-brass for the bcc-phase. The peak shift of the copper alloy (fcc phase) compared to 1+2 is clearly visible in the diffraction patterns. The ‘normal shot’ went through 3+4, and confirms results on point P3+P4 Lattice parameter of bronze/brass: a=3.686 Å. This value can be interpreted as $Cu_{0.7}Zn_{0.3}$ brass (= 40wt% Zn) or a bronze $Cu_{0.93}Sn_{0.07}$ (= 12wt% Sn). These cases are indistinguishable. - no filling materials	none	 - The springs are made of Cu and brass. The brass consists of $Cu_{64}Zn_{36}$, with alpha-brass on the surface and beta-brass in there wire core. TOF-ND identifies the different phases of the two types of springs: two Cu-type phases with different lattice parameters. - TOF-ND also **identifies** the beta-brass component (*bcc*-phase) which is consistent with the Cu-Zn phase diagram for a $Cu_{60}Zn_{40}$ brass.

Table 2 (a). *TOF Neutron Diffraction results on D-IV*

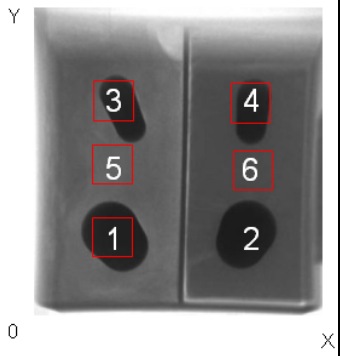
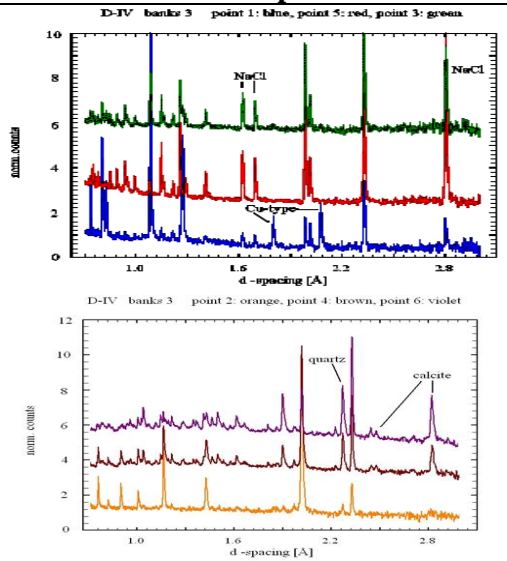
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
D-IV		**Instrument: ROTAX** beam size 10x10 mm; beam along z box alignment: P1-P6 aligned with incident beam; no offset		**Point 1**: NaCl, Cu-type (fcc) a=3.592 Å, steel (Fe) or copper (Cu) **Point 2**: bcc a=2.845 Å, ferrite (Fe) + cementite (Fe_3C), in clay=calcite+quartz **Point 3**: NaCl, small fcc peaks a=3.6 Å, steel (Fe) or copper (Cu) **Point 4**: calcite, quartz, bcc a=2.845 Å, ferrite (Fe) **Point 5**: NaCl **Point 6**: calcite (75wt%), quartz (25 wt%)

Table 2 (b). *Comments on TOF-ND results on D-IV*

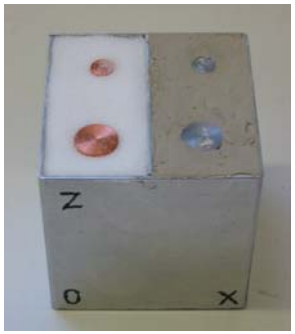
Box no.	Box content from TOF-ND	Complementary info	Reality Check
D-IV	- Two compartments with different filling materials: salt (NaCl) and clay consisting of quartz (25 wt% SiO_2) and calcite (75wt% $CaCO_3$). - The **objects** in the two chambers are made of different materials: **1**: fcc-structure, Cu-type (fcc structure); lattice parameter (a=3.592 Å) is closer to steel (3.594 A) than to copper (3.615 A) **2**: Bcc lattice, ferrite (Fe); extra cementite (Fe_3C) peaks; clay peaks are small compared to the Fe peaks; **3**: fcc-structure, Cu-type (fcc structure); lattice parameter (a=3.592 Å) indicates steel **4**: The rod material is bcc-Fe (ferrite)	- **PGAA** identifies fcc-metal as **Cu** for points (1) and (3) - **PGAA** identifies **Al** as separator material. 	Cu and Iron rods, embedded in halite (NaCl) and clay (51% calcite, 20% quartz, 12% muscovite, 17% kaolinite) - The main components are identified by TOF-ND. - PGAA is required to decide on the fcc-material: copper. - For the clay the two main components were identified with approximately the correct proportions. - After disclosure of the content, kaolinite is identified in the pattern. - Muscovite was not detected by TOF-ND. - The lattice parameters of Cu and Fe are systematically shifted towards lower values. This is probably due to absorption (i.e. apparent shift of the material towards the neutron source.) - **Extra phase**: Cementite is observed in the ferrite. - The second wall of the box is not visible for both filling materials. The Cu rod in position 3 was almost missed by the neutron beam due to misalignment of the box on the instrument.

Table 3 (a). *TOF Neutron Diffraction results on D-V*

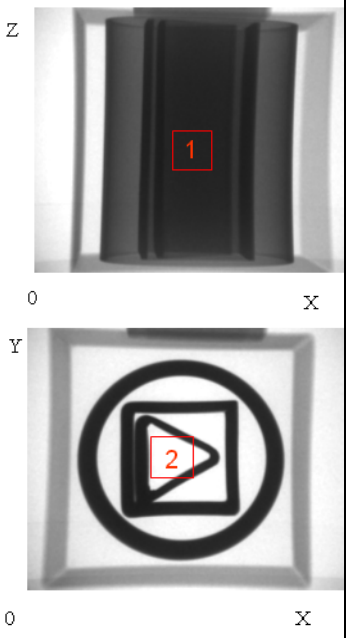
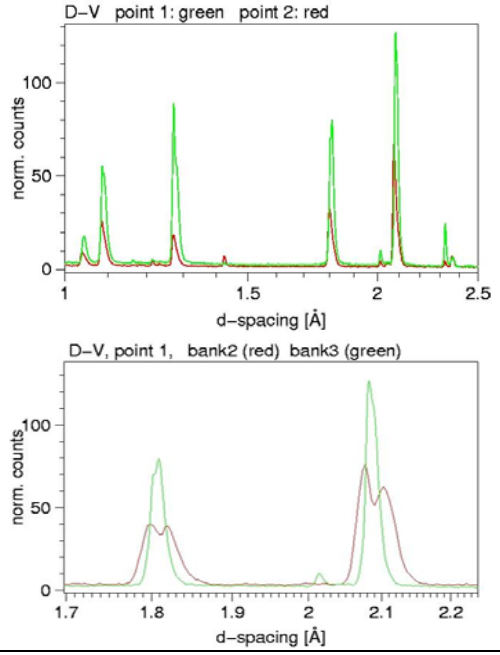
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
D-V		**Instrument: ROTAX** Point 1: beam size 10x10 mm; beam along y Point 2: beam size 10x10 mm; beam along z box alignment: P1-2 aligned with incident beam; no offset		**Point 1**: Cu-type (*fcc*), a=3.608 Å **Point 2**: Cu-type (*fcc*), a=3.598 Å

Table 3 (b). *Comments on TOF-ND results on D-V*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
D-V	- The two analysis points show the **same set of peaks**, i.e. all the parts of the internal object are made of the **same material**; - The object is made of an fcc material with **Cu type structure**; the lattice parameters point to steel rather than copper but peaks can be shifted due to displacement of the object inside the box. - The rest of the **box** is **empty**. - Inspection of the diffraction patterns shows the following features: **1.** Fcc Cu-type material is textured (preferred orientation of grains) **2.** The aluminium peak intensities are different for point 1 and point 2 due to absorption of neutrons in the copper **3.** Splitting of peaks in bank-2 (double pattern) indicates transition of beam through two walls.	- **PGAA** identifies fcc-metal as **Cu**.	 - The **objects** consist of **pure copper**. - The main phase is identified by TOF-ND. PGAA differentiates between steel and Cu.

Table 4 (a). *TOF Neutron Diffraction results on D-VI*

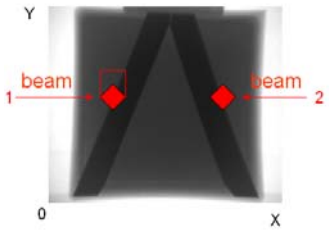
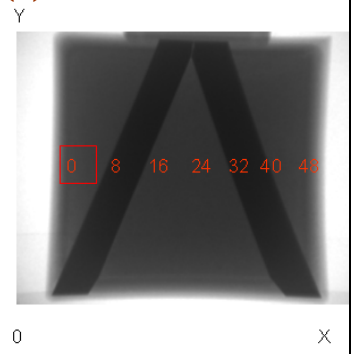
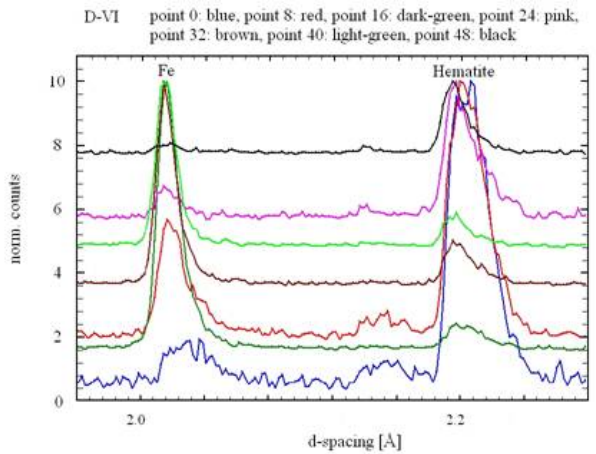
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
D-VI	**(a)**  **(b)** 	**Instrument: ROTAX** **(a)** Point 1: beam size 10x10 mm; beam along x Point 2: beam size 10x10 mm; beam along minus x **(b)** Scan along x [mm]; beam size 10x10 mm; beam along z box alignment: no offset		**(a)** **Point 1**: Hematite (Fe_2O_3), bcc type structure, a=2.86 Å, ferrite (Fe) **Point 2**: Hematite (Fe_2O_3) , bcc type structure, a=2.85 Å, ferrite (Fe) **(b)** **Point 0**: Fe_2O_3 (Hematite), wall **Point 8**: Fe_2O_3 (17wt%), ferrite (83wt%) **Point 16**: Fe_2O_3 (86 wt%), ferrite (14 wt%) **Point 24**: Fe_2O_3 (94 wt%), ferrite (6 wt%) **Point 32**: Fe_2O_3 (38wt%), ferrite (62wt%) **Point 40**: Fe_2O_3 (37wt%), ferrite (63wt%) **Point 48**: Fe_2O_3 (100wt%) ferrite (0 wt%)

Table 4 (b). *Comments on TOF-ND results on D-VI*

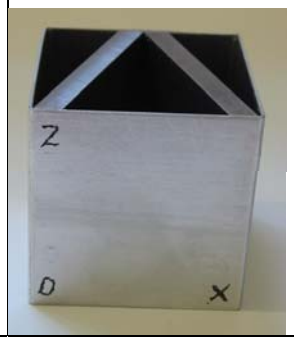
Box no.	Box content from TOF-ND	Complementary info	Reality Check
D-VI	- The two inclined '**plates**' are of the **same material**: ferrite (bcc-Fe); - The x-scan shows that the V-shape object is **ferrite** and that the box is **filled with hematite** (not the other way round).	- **PGAA** confirms presence of **Fe** and absence of other elements;	 - The main components (ferrite and hematite) are identified. - This box highlights the **advantage** of **TOF-ND** compared to element-sensitive methods: oxides, corrosion phases, secondary alteration phases can be distinguished from alloys.

Table 5 (a). *TOF Neutron Diffraction results on D-VII*

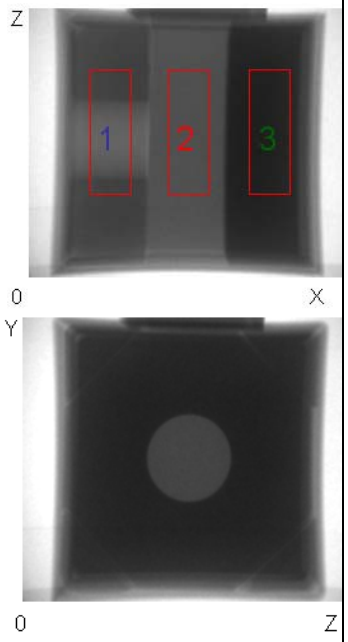
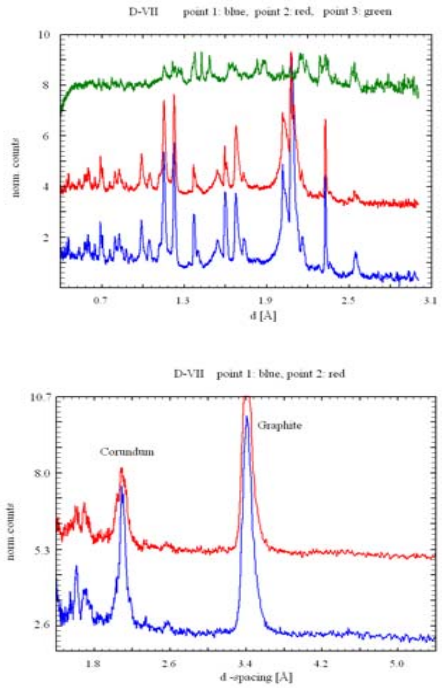
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
D-VII		**Instrument: ROTAX** beam size 10x30 mm; beam along y box alignment: no offset		**Point 1**: graphite (C) + corundum (Al_2O_3) in equal parts **Point 2**: graphite (C) + corundum (Al_2O_3) in almost equal parts **Point 3**: phase not identified

Table 5 (b). *Comments on TOF-ND results on D-VII*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
D-VII	- Analysis **points 1** and **2** are made of the **same material**: graphite and corundum. - Analysis point 3 shows a rich spectrum of diffraction peaks. The pattern could not be unravelled, or assigned to a single phase.	- **PGAA** identifies **Na** which may come from diaoyudaoite ($Al_{10.35}$ $Mg_{0.65}O_{16}$)($Na_{1.65}O$) - **PGAA** identifies **Si** and **H** which comes from pyrophyllite	 - **Graphite** and **corundum** were **identified**. - The peaks of diaoyudaoite ($Al_{10.35}$ $Mg_{0.65}O_{16}$)($Na_{1.65}O$) at 11.2 and 5.7 Å were observed but the compounds was not identified. - The peak-rich pattern of pyrophyllite was recognised, but the mineral was not identified. - The simultaneous observation of graphite and corundum in equal amounts for point 1 and point 2 indicates a substantial **misalignment** of the box on the diffractometer, or wrong axes description on the radiographies.

Table 6 (a). *TOF Neutron Diffraction results on D-VIII*

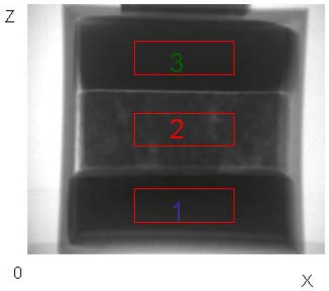
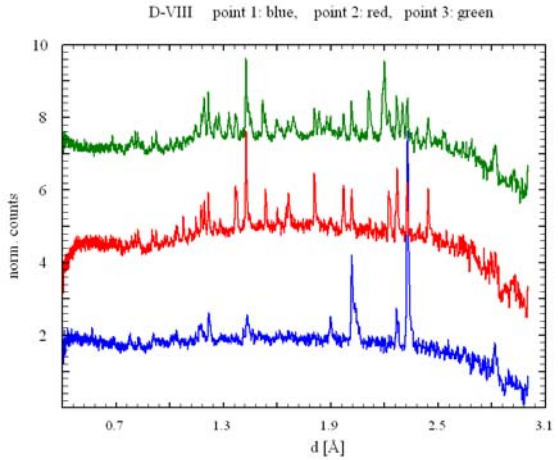
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
D-VIII		**Instrument: ROTAX** beam size 30x10 mm; beam along y box alignment: no offset		**Point 1**: calcite ($CaCO_3$), high background **Point 2**: quartz (SiO_2), on high amorphous background **Point 3**: quartz (SiO_2)(25wt%), mullite $3Al_2O_3$ $2SiO_2$ (55wt%), cordierite $Mg_2Al_4Si_5O_{18}$ (10wt%), cristobalite (SiO_2) (5wt%), identified in the data after box content is revealed; other un-indexed peaks on high background level

Table 6 (b). *Comments on TOF-ND results on D-VIII*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
D-VIII	- **Point 1** shows a very high hydrogen background indicating mineral with H-content; the beam is stopped in the box, there are no Al-peaks from the back aluminium wall of the box; there are practically no peaks in forward scattering. The main component is calcite. - In **Point 2** quartz is identified. - **Point 3** has a large amorphous background, most likely due to H-containing substance/mineral, or glassy component. The patterns contain Bragg peaks from quartz, mullite, cordierite and cristobalite. A large peak at 2.84 A is unidentified.	- **PGAA** identifies **hydrogen** on all points, in agreement with the neutron background, in contrast to x-ray analysis.	 - **P1** is on a slab of clay. TOF-ND sees the main component, calcite. The clay seems to be more hydrogenous than in D-IV. - **P2** is on a slab of brick stone (quartz, albite, muscovite, hematite). TOF-ND sees only main component, quartz. - **P3** is on a slab of fire brick (schamotte) (mullite, quartz, cordierite). The phases mullite, cordierite were not identified from the observed peak-rich spectrum because the peaks were only from backscattering. (Mineral phases are best identified in forward scattering). Once the box content was revealed, the phase fractions from TOF-ND are in the right order of magnitude.

Table 7 (a). *TOF Neutron Diffraction results on D-IX*

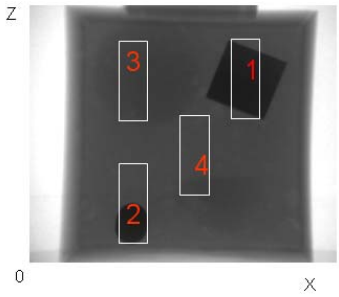
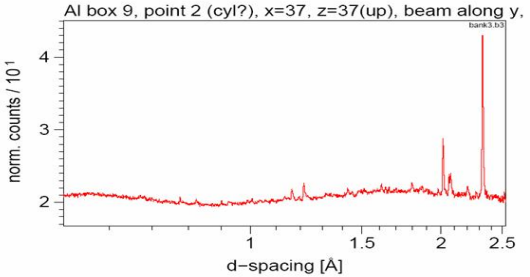
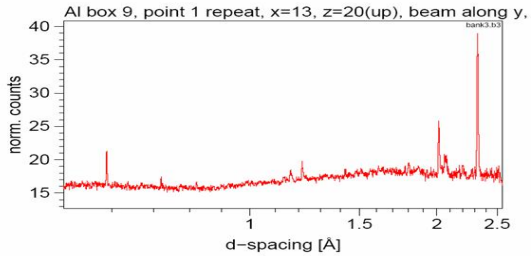
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
D-IX		**Instrument: ROTAX** beam size 10x30 mm; beam along y box alignment: P1-P4 aligned with incident beam; offset on diffractometer according to radiography	 	**Point 1-4**: gypsum, high amorphous background

Table 7 (b). *Comments on TOF-ND results on D-IX*

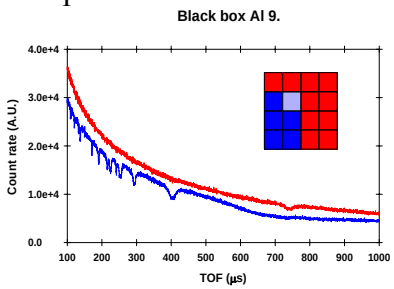
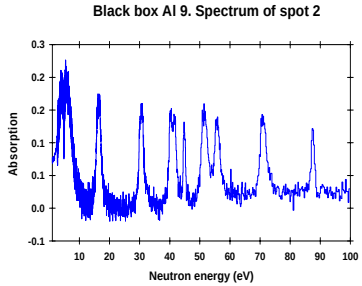
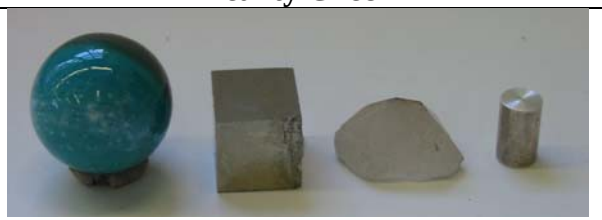
Box no.	Box content from TOF-ND	Complementary info	Reality Check
D-IX	- All patterns exhibit a very **high** (hydrogen) **background**, i.e. indicate compounds with hydrogen. - The patterns only show the front wall of the Al-cube, i.e. the beam is stopped within the box. - There are **weak peaks** in the patterns from the filling material (they are in all patterns). The filling material is not identified. - **P1** has extra single crystal peaks in backscattering at low d-spacing; not possible to index with low d-value lines;	- **NRCA** analysis on INES identifies the Ag rod on point 2.  	 - Box is filled with gypsum ($CaSO_4$ $(H_2O)_2$). - Embedded are: **1.** pyrite single crystal (FeS2) in point 1 **2.** Silver rod in point 2 **3.** Glass sphere in point 3 **4.** Quartz crystal in point 4 - The extra peaks of the filling material are only identified after disclosure of the box fillings. - The single crystal peaks of FeS_2 are visible in the data but were not identified. - no identifiable signals from the other objects in the box. The glassy signal from the glass object is covered by the gypsum scattering. Ag-peaks in TOF-ND data are hidden under the Al peaks.

Table 8 (a). *TOF Neutron Diffraction results on D-X*

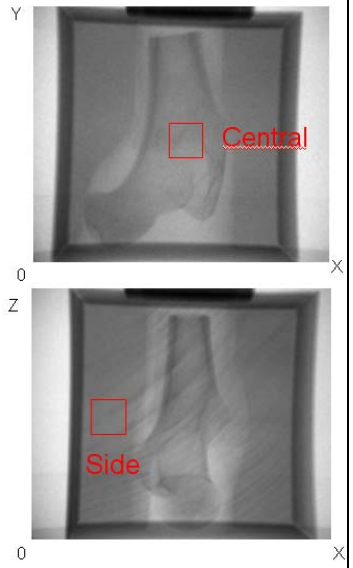
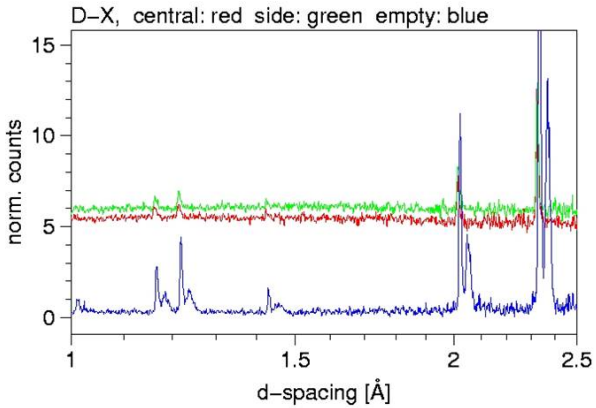
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
D-X		**Instrument: ROTAX** beam size 10x10 mm; box alignment: no offset		**Central**: beam along z, high background scattering **Side**: beam along y, high background scattering

Table 8 (b). *Comments on TOF-ND results on D-X*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
D-X	- The diffraction patterns do show Al peaks from the box wall, but only from one wall. - - The aluminium peaks are only in backscattering; - Therefore the box cannot be empty. There are no peaks of bone material. - The high neutron background indicates scattering by hydrogen; the hydrogen can be in the bone (visible in x-ray radiography) but there may also be filling material around the bone;	none	- Badger bone, inside beech wood, lower part of bone wrapped in leather; - TOF-ND cannot provide information due to missing crystallinity of the material.

Table 9 (a). *TOF Neutron Diffraction results on D-XI*

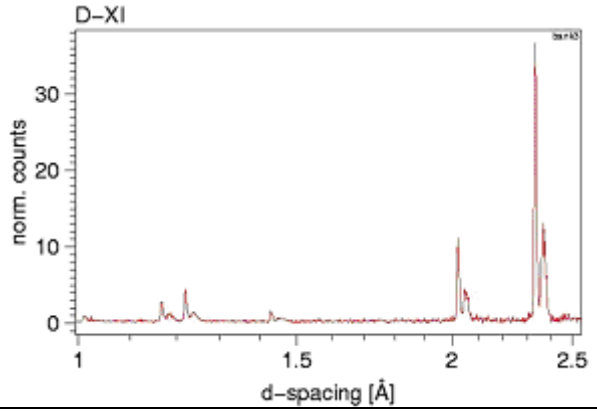
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
D-XI		**Instrument: ROTAX** beam size 10x30 mm; box alignment: no offset		Doublets of Al peaks from front and back-wall scattering

Table 9 (b). *Comments on TOF-ND results on D-XI*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
D-XI	- We observe diffraction patterns from the **front wall** and from the **back wall**. The patterns show the fcc structure of aluminium. Moreover, the patterns show a drastic texture (preferred grain orientation of the Al grains). - The **distance** between the **double peaks** is related to the distance between front and back wall of the box (x): $\Delta d/d = x * \cos(\theta) / L_2$ where Δd is the distance between two peaks in Å, L_2 the distance between sample position and detector, and where θ is ½ the detector angle. - The **intensities** of the **peaks** from the back wall are much reduced compared to the peak intensities from the front wall due to absorption.	none	- **Empty box**, made of aluminium. The front and back walls are x=50 mm apart. Wall thickness 1 mm.

Table 10 (a). *TOF Neutron Diffraction results on H-I*

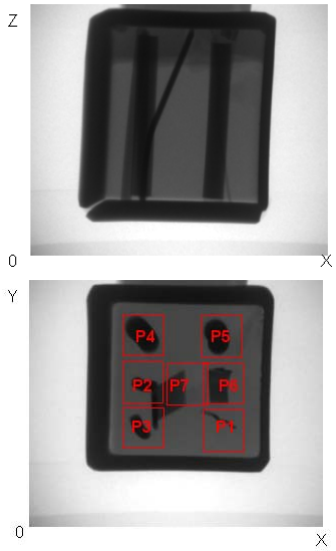
Box no.	X-ray radiograph	Set-Up	Diffraction patterns
H-I		**Instrument: GEM** beam size10x10 mm; beam along z (top side towards beam, side with number "I" facing down) box alignment: no offset	**P1**: copper or steel (small peaks) **P2**: copper a=3.62 Å, small copper alloy component a=3.7 Å, Al/Ag **P3**: copper alloy a=3.69 Å **P4**: fcc-phase a=3.7 Å: bronze or brass; Al/Ag **P5**: copper alloy, a=3.62 Å, Al/Ag, maybe Zn **P6**: fcc a=3.60 Å (copper or steel), Al/Ag **P7**: fcc a=3.60 Å (small peaks) All diffraction patterns contains gypsum ($CaSO_4(H_2O)_2$) peaks.

Table 10 (b). *Comments on TOF- ND results on H-I*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
H-I	The analysis points P1-7 reveal the following main materials (in addition to gypsum): **P1:** copper (Cu) or steel (Fe) **P2:** copper **P3:** brass or bronze **P4:** brass or brass **P5:** copper, indications of Zn; **P6:** copper or steel; strong Al/Ag peaks **P7**: indications of copper/steel peaks - All patterns show Al/Ag peaks, apart from P1, P6	- **PGAA** observed **Cu**, **Zn** and **Fe**, i.e. no Sn. This means, the copper alloy is most likely brass (Cu-Zn). - **PGAA** does not find Al or Ag.	 **1** - copper wire **2** - brass wire **3** - copper wire **4** - brass rod **5** - copper rod **6** - zinc plate **7** - iron plate on gypsum plate - Copper on **P1** was first overlooked, but peaks are present in data. **P1**, **P4**, **P5** were assigned correctly. - **P2**, **P3** analyses are wrong. **P2** was analysed as copper; small brass component is correct. **P3** was analysed as brass, but it is copper. Since copper and brass are easy to distinguish there seems to be an alignment error. Zinc on **P6** is not identified (Zn: peaks at 2.09, 2.31, 2.47 Å). There are small Zn peaks in **P2**. - **P7** shows fcc-phase, in agreement with steel. Pb peaks, identified in the first analysis, are from gypsum. - Ag/Al was identified but is not present.

Table 11 (a). *TOF Neutron Diffraction results on H-II*

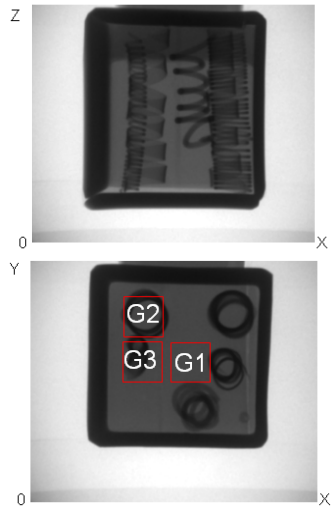
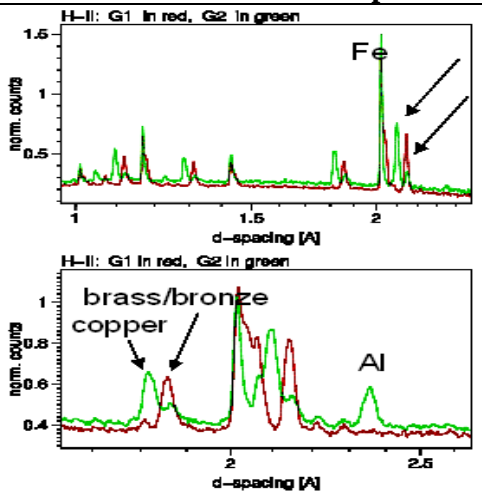
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
H-II		**Instrument: GEM** beam size10x10 mm; beam along z box alignment: no offset	 copper brass/bronze	"**Filling**" peaks gypsum: $CaSO_4(H_2O)_2$ **G1:** gypsum, strong copper alloy peaks (a=3.70 Å) **G2**: gypsum, strong copper/steel (a=3.62 Å), + weak copper alloy (a=3.70 Å) peaks; **G3**: gypsum, Al, copper alloy/steel phase (a=3.59 Å), strong Al/Ag

Table 11 (b). *Comments on ND results (Black Box H-II)*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
H-II	- The **filling material** is gypsum. The box contains metallic springs, made of different materials: copper/steel and bronze/brass. - **G1** in the centre shows strong copper alloy peaks (bronze or brass), presumably from one of the springs. - **G2** shows a copper/steel fcc-phase, next to a much weaker bronze/brass phase. Extra strong peaks indicate Al/Ag. - **G3** shows a weak copper/steel phase. Extra strong peaks indicate Al/Ag. - The Cu/Al phases are not present in the forward scattering banks.	none	 **1** - brass spring **2** - brass spring **3** - copper spring **4** - brass spring **5** - copper spring - The springs were correctly identified as metal (bronze or brass) springs. - The points **G1** was intended as a shot in the centre to determine the filling material. The presence of copper in the data indicates a "failed shot". - **G2** (brass) is wrongly assigned as copper. **G3** (Cu) is correct. - Beta brass components are not detected. - Gypsum was not identified as a plate, but as a powder filling. This could have been detected by comparing the patterns to the ones of other gypsum-filled boxes. There could be a mis-orientation of the box on the instrument.

Table 12 (a). *TOF Neutron Diffraction results on H-III*

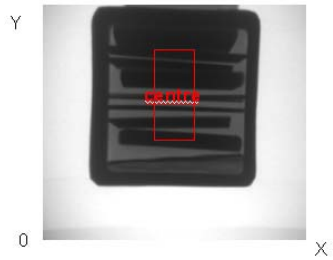
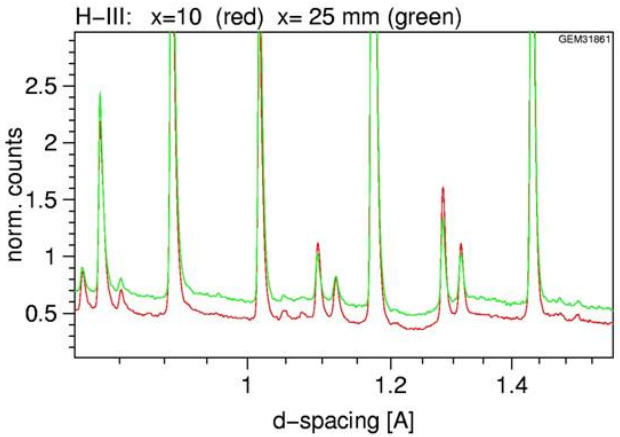
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	ND results
H-III	**(a)**  **(b)** 	**Instrument: GEM** **(a)** beam size 5x20 mm; beam along y (side with number H-III facing incoming beam) **(b)** Beam size 10x20 mm; beam along z box alignment: no offset		**(a)** **Point 1**: x=5, copper alloy 1 (a=3.618 Å), copper alloy 2 (a=3.70 Å) **Point 2**: x=10, copper alloy 1 (a=3.614 Å), copper alloy 2 (a=3.70 Å) **Point 3**: x=15, copper alloy 1 (a=3.613 Å), copper alloy 2 (a=3.70 Å) **Point 4**: x=20, copper alloy 1 (a=3.613 Å), copper alloy 2 (a=3.70 Å) **Point 5**: x=22.5, copper alloy 1 (a=3.613 Å), copper alloy 2 (a=3.70 Å) **Point 6**: x=25, copper alloy 1 (a=3.613 Å), copper alloy 2 (a=3.70 Å) **Point 7**: x=30, copper alloy 1 (a=3.613 Å), copper alloy 2 (a=3.70 Å) **Point 8**: x=32.5, copper alloy 1 (a=3.613 Å), copper alloy 2 (a=3.70 Å) **(b)** **Point Centre**: copper alloy 1 (a=3.615 Å), copper alloy 2 (a=3.70 Å)

Table 12 (b). *Comments on TOF-ND results on H-III*

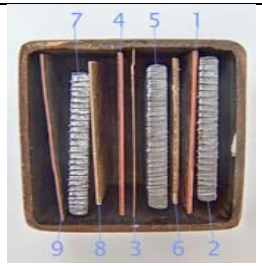
Box no.	Box content from TOF-ND	Complementary info	Reality Check
H-III	- The data show the same phases (copper-type phase, copper alloy phase) for all points, in similar phase ratios. - The **copper-type phase** (a=3.613 Å) can be steel or copper. - The copper alloy (a=3.7 Å) can be bronze or brass. - There is **no filling material** identified.	- **PGAA** data show copper and iron as elements. The PGAA scan identifies the copper-type phase as copper. No filling material is detected. - **PGAA** identifies the copper alloy with the larger lattice parameter as brass.	 **1** - copper sheet **2** - iron sheet **3** - brass sheet **4** - copper sheet **5** - iron sheet **6** - brass sheet **7** - iron sheet **8** - brass sheet **9** - steel sheet - All TOF-data show the same pattern. It has to be assumed that the scan was not, as planned, across the different plates. Maybe the incoming beam was impinging on the flat side of the sheets. - TOF-ND does not (easily) recognise an iron object in an iron box, although it is principally possible to separate the inside-objects via peak shift analysis. - TOF-ND failed to identify the steel sheet (9).

Table 13 (a). *TOF Neutron Diffraction results on H-IV*

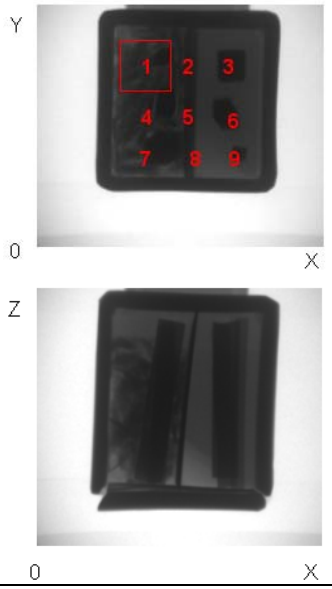
Box no.	X-ray radiograph	Set-Up	Diffraction patterns
H-IV		**Instrument: GEM** beam size 10x10 mm; beam along z (top side facing incoming beam, side with numbers down) box alignment: no offset	All patterns exhibit: quartz (SiO_2), gypsum $CaSO_4(H_2O)_2$, copper alloy (a=3.654 Å), **Point 1**: Quartz (12%), gypsum (7%), copper alloy (3%), Al/Ag (1%), FeO (3%), Fe_3O_4 **Point 4**: Quartz (17%), gypsum (5%), copper alloy (2%), FeO (2%), Fe_3O_4 **Point 7**: Quartz (11%), gypsum (6%), copper alloy (2%), FeO (2%) **Point 2**: Quartz (26%), gypsum (7%), copper alloy (3%), Al/Ag (5%), FeO (1%) **Point 5**: Quartz (27%), gypsum (7%), copper alloy (3%), Al/Ag (2%) **Point 8**: Quartz (17%), gypsum (7%), copper alloy (3%) **Point 3**: Quartz (31%), gypsum (6%), copper alloy (3%), Al/Ag (13%) **Point 6**: Quartz (27%), gypsum (6%), copper alloy (2%), Al/Ag (12%) **Point 9**: Quartz (20%), gypsum (5%), copper alloy (3%))

Table 13 (b). *Comments on TOF-ND results on H-IV*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
H-IV	- The box is divided into **two parts**. All data show quartz and gypsum. However, P3, P6, P9 contains much more quartz. - The **copper alloy** can be brass (17wt% Zn) or bronze (5 wt% Sn). This component is high for P2,5,8, and low otherwise, indication that the separating sheet is made out of copper alloy. - **Copper alloy** contents are small.	- **PGAA** idenfies Fe and Mn, with the latter probably in the ferrite. - **PGAA** identifies Al, rather than Ag. - One filling material contains Si and B (?) as main component (SiO_2). The other filling material contains Fe as main component. - **PGAA** identifies Cu, in agreement with the TOF-data.	 **1**, **2**, **3** - iron **4** - thin steel plate **5** - fill grit **6** - fill sand - TOF-ND does not easily recognise a ferrite object in a box made of ferritic iron, although it is principally possible to separate the inside-objects via peak shift analysis from the wall material. - The gypsum was not identified as a plate, but as a powder filling. Has quartz spilled into left side of the box? Where is Al and Cu in the box? The nominal elemental composition is: Fe, Mn, C, B, Al, Si, K, Ti

Table 14 (a). *TOF Neutron Diffraction results on H-V*

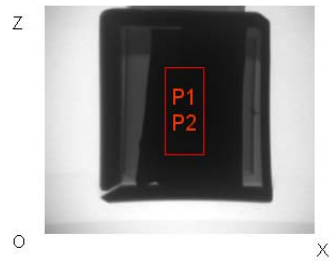
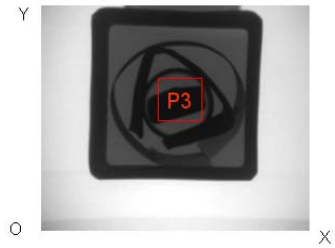
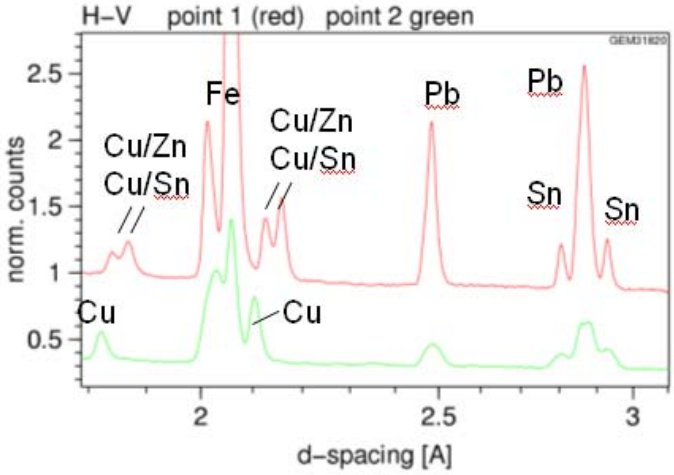
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	Box no.
H-V	**(a)**  **(b)** 	**Instrument: GEM** **(a)** beam size 10x20 mm; beam along y **(b)** beam size 10x10mm; beam along z box alignment: no offset		**(a)** **P1+2**: gypsum $CaSO_4$ $(H_2O)_2$ (small peaks), Pb, Sn, copper alloy (a=3.7 Å) **(b)** **P3**: gypsum CaSO4 (H2O)2 (strong peaks), Pb, Sn, copper alloy (a=3.63 Å), Al/Ag (small)

Table 14 (b). *Comments on TOF-ND results on H-V*

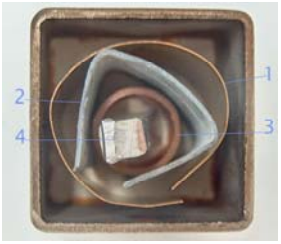
Box no.	Box content from TOF-ND	Complementary info	Reality Check
H-V	- The box contains gypsum (filling). - There is a combination of metals: Pb, Sn, copper alloy, Al/Ag. - **P1** shows split bronze/brass peaks, but **P3** not. Therefore the bronze/brass might be the outermost ring; the copper alloy peaks are different for P1 and P3. - The Pb peaks are split in P2. Therefore Pb could be the second-outermost metal in the box. The Sn could be part of a Pb/Sn solder. - There is a **peak** at **2.34 Å** in P3 which indicates a fcc metal such as Al or Ag/Au. Pb and Sn are present in the center as well. - **P3** shows the 2.34 Å peak while it is hardly visible in P1. Therefore is can be assumed that Al/Ag/Au is the innermost metal in the box.	none	 **1** - brass plate **2** - lead plate **3** - copper tube **4** - thin lead solider Mounted on a gypsum plate - The outer ring was correctly identified as copper alloy. Cu/Sn and Cu/Zn cannot be distinguished by TOF-ND. - The Pb ring was correctly identified. - The different copper alloy for P3 corresponds to the (short) copper ring which is out of sight in P1. The Cu lattice constant from TOF-ND is too large. - The inner metal is Pb/Sn eutectic which gives separate Pb and Sn peaks. Pb/Sn inside a Pb-sheet is difficult to see. - Al/Ag was wrongly identified as the centre object. Is there a top Al plate? - The gypsum peaks are smaller in P1+2 compared to P3 because there is just the bottom gypsum plate. Gypsum peaks in P3 remain unexplained

Table 15 (a). *TOF Neutron Diffraction results on H-VI*

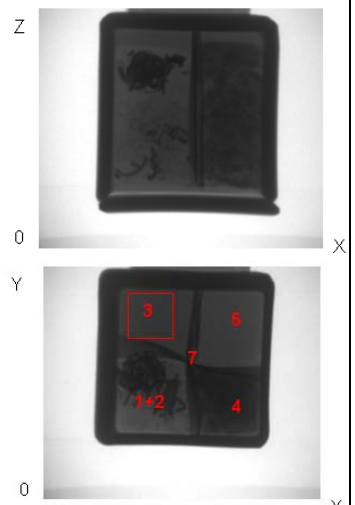
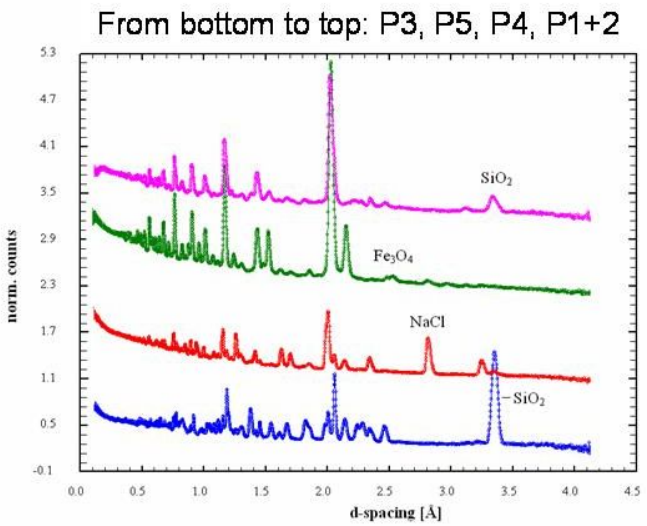
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	Box no.
H-VI		**Instrument: GEM** beam size 10x10 mm; beam along z box alignment: no offset		**P1+2:** quartz (SiO_2), gypsum ($CaSO_4(H_2O)_2$), talc ($Mg_3(OH)_2(Si_4O_{10})$), Al or Ag **P3:** quartz, gypsum, talc, Al/Ag, fcc-Cu-type (a=3.7 Å) **P4:** gypsum, talc, fcc-Cu-type (a=3.7 Å), Magnetite (Fe_3O_4), maybe FeO; **P5:** quartz, gypsum, halite (NaCl), gypsum, talc, fcc Cu-type (a=3.7 Å) **P7:** gypsum, halite, talc, fcc Cu-type (a=3.7 Å), Al/Ag, FeO

Table 15 (b). *Comments on TOF-ND results on H-VI*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
H-VI	- The radiography shows that the box is divided into 4 sectors. - There are a number of peaks that are common to **all sectors**, either because it is a filling material or a wall material: gypsum, Al, talc (peaks at 9.6, 4.56 3.13 Å). - There is the following additional material in the *4 chambers*: • Main phase in **P1+2**: Al/Ag • Main phase in **P3**: quartz • Main phase in **P5**: halite (NaCl) • Main phases in **P4**: iron oxides (FeO, Fe_3O_4)	- **PGAA** identifies Ag rather than Al. - **PGAA** confirms the presence of a copper alloy as the fcc-phase. - The presence of Zn indicates brass rather than bronze.	 **1** - silver wire **2** - fill talcum **3** - fill sand **4** - fill grit iron **5** - fill salt (NaCl) **6** – copper sheets **7** –aluminium sheet - The main components in the compartments were identified. - The fcc-copper phase has a lattice parameter that rather indicates brass than pure copper, confirmed by PGAA. - The brass sheets are visible in all data except for point 1+2 (larger sector) which indicates a slight problem with the alignment of the box. - TOF-ND shows talc in all sectors. This result may indicate a misalignment or it may indicate that talc powder leaked from 1+2 into other compartments

Table 16 (a). *TOF Neutron Diffraction results on H-VIII*

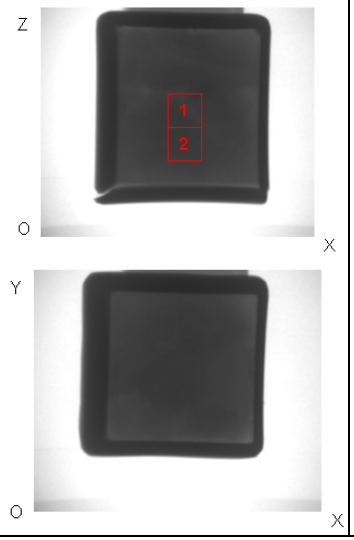
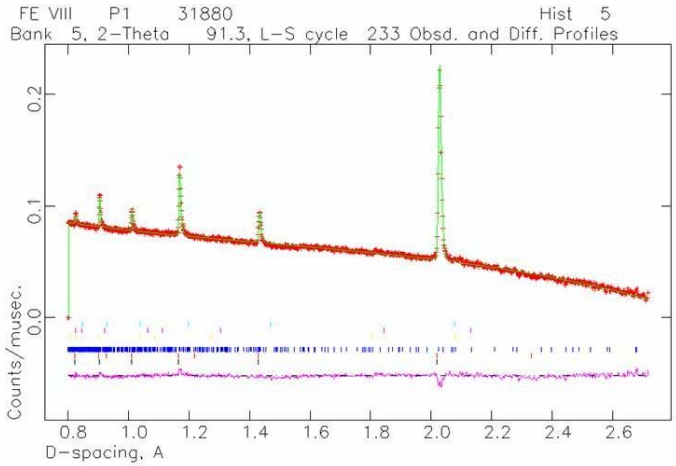
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	Box no.
H-VIII		**Instrument: GEM** Beam along y; side with number "VIII" facing incoming beam box alignment: no offset		All diffraction patterns show single Fe peaks from the box walls. **Point 1**: moderate background **Point 2**: moderate background

Table 16 (b). *Comments on TOF-ND results on H-VIII*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
H-VIII	- Only Bragg peaks of ferrite (front wall) are observed. - The beam does not pass through the box. - The box is filled with a neutron-stopping material. - TOF-ND "sees" no crystalline material. - The backgrounds in the neutron diffraction patterns are decreasing with increasing d-spacing (i.e. with increasing wavelengths). This is typical for absorbing or self-absorbing material (e.g. hydrogen-containing compounds).	- **PGAA** data suggest gypsum as main material (Ca, S, H). - Other elements from **PGAA** are Al, Si, K, Ti. .	  - Neolitic chaff tempered pottery; filling material is gypsum - The material inside the box is not identified. There are slight hints of gypsum peaks in the TOF-ND data. - The gypsum filler is impenetrable for the thermal neutron beam.

Table 17 (a). *TOF Neutron Diffraction results on H-IX*

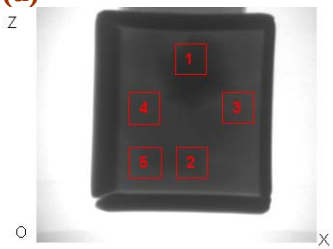
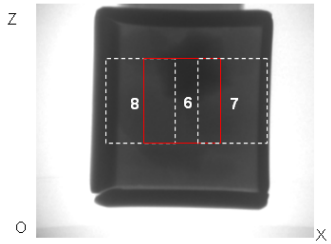
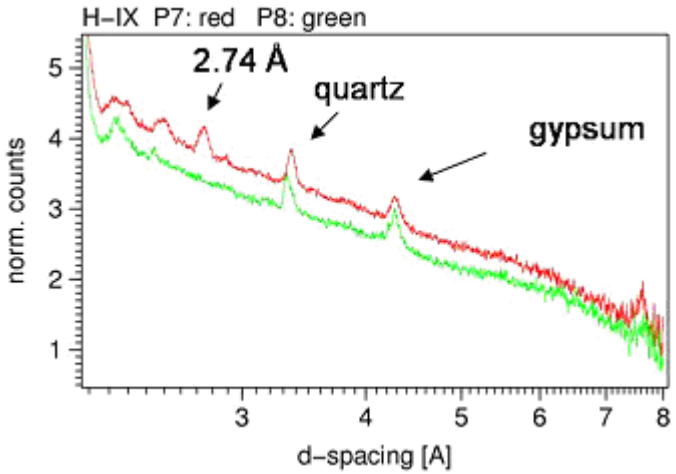
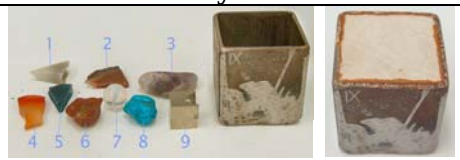
Box no.	X-ray radiograph	Set-Up	Diffraction patterns	Box no.
H-IX	**(a)**  **(b)**  	**Instrument: GEM** Beam along y (side with number "IX" facing incoming beam) **(a)** Beam size: 10x10 mm **(b)** Beam size: 20x40 mm box alignment: no offset	 	Diffraction patterns show strong Fe peaks from front wall and weak peaks from back wall of the box. All points P1-P8 show gypsum $CaSO_4(H_2O)_2$ and quartz (SiO_2). **(a)** **P1**: extra characteristic peaks at 2.33, 3.31 Å **P2**: extra characteristic peaks at 2.50, 2.73 Å **P3**: extra characteristic peaks at 2.33, 2.73 Å **P4**: extra characteristic peak at 2.33 Å **(b)** **P6**: extra characteristic peaks at 2.44, 2.73, 3.31 Å **P7**: extra characteristic peak at 2.73 Å

Table 17 (b). *Comments on TOF-ND results on H-IX*

Box no.	Box content from TOF-ND	Complementary info	Reality Check
H-IX	- Apart from Fe peaks, there is a confusing pattern of Bragg peaks. All points show quartz and gypsum peaks (mixture of the two as filler). - **Gypsum** peaks are observed in forward scattering. This means that the neutrons are able to penetrate the box, in contrast to H-VIII. - The background in the neutron diffraction patterns is decreasing with increasing d-spacing (i.e. with increasing wavelengths). This is typical for a hydrogen-containing material, such as the gypsum filler). - 2.33 Å peaks in P1,3,4 indicate presence of Al/Ag.	none	 ***1, 2*** *– achat* ***3*** *- amethyst* ***4, 6*** *– cornean* ***5*** *- blue glass* ***7****-achat globular bead* ***8****–turqoise* ***9*** *-pyrite* **Filler material**: gypsum - **Gypsum** and **quartz** are identified. Gypsum is the filling material, quartz is present in form of several localized objects rather than a filler. A finer scan would have revealed this. - **Achat** and **amethyst** are different varieties of quartz (SiO_2). - What's cornean? - The glass bead was not detected. - P2,3,6,7 show pyrite (2.74 Å peak). This does not agree with the cube-feature in the radiography. (orientation problem?) - Al/Ag is not present. The peaks at 2.33, 2.44, 3.31 Å are unexplained. - Quartz in P5 is polycrystalline, while quartz in P1 is a single crystal. - It is surprising that this box is more transparent than H-VIII.