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Proficiency testing of used cold rolling emulsion and fresh oil concentrate

Anders Janols

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Cold rolling, emulsion, lubrication, laboratory, chemical analysis, proficiency test

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Anders Janols, SSAB EMEA Borlänge

Summary

In November 2009 eight laboratories from four European countries carried out a proficiency test on used cold rolling mill emulsion and fresh oil concentrate used to make up this emulsion. One main purpose of this initiative was to verify the results regarding the suitablilty for performing proficiency testings with such emulsions achieved from two minor Round Robins among four Nordic labs and also a methods study made by SSAB to investigate the impact on analysis results of longer times for transport and the recommended routines for preparing the samples before analysis work.

An important objective was also to gain knowledge of distribution of results between laboratories and methods, in case the proficiency tests on these types of samples were assessed reliable.

From the samples of used mill emulsion the following variables were investigated: oil concentration, saponification number, total acid number, ashes, iron tot and pH.

Fresh oil concentrate were analysed for saponification number, total acid number and kinematic viscosity (40˚C).

Compared to the measurement uncertainty at SSAB (coordinating lab) the results of this proficiency testing was in accordance with what could be expected, with respect to significant differences of methods for some of the variables.

With applied routines for the realization of this testing, it has been proved that it is possible to compare used cold rolling mill in proficiency testings.

Proficiency testing of used cold rolling emulsion and fresh oil concentrate

Anders Janols, SSAB EMEA Borlänge

In used emulsion from a cold rolling mill *oil content, saponification number (SAP), total acid number (TAN), ashes, total iron (Fe tot)* and *pH* were determined from double samples. Also fresh oil concentrate from the batches present in emulsion oil at the time of sampling was tested for *SAP*, *TAN* and *kinematic viscosity (40˚C)* in doublets.

Methods study on used emulsion

Since cold rolling emulsions in many aspects are highly dynamic systems, they have not earlier been the subjects for proficiency testings. Within Jernkontoret (JK), The Swedish Steel Producers Association, two minor Round Robins on used cold rolling emulsion and fresh oil concentrate have been performed in 2007 and 2008 with satisfactory results. For that reason the committee of JK on analysis of process chemicals decided to investigate the suitability of a proficiency test with a higher number of participants.

A methods study with the purpose of investigating the impact on analysis results from method of sampling, storing/transport and handling of used emulsion before analysis work was made in May 2009 at the chemical laboratory of SSAB EMEA, Borlänge plant.

The summary of the preliminary study is presented in Annex 1-1, the results in Annexes 1-2 and 1-3. Method for sampling is described in Annex 2 and suggestion for transport and lab work in Annex 3. In Annex 4 the analysis methods used at the study in SSAB are compiled.

After comparing the result with the documented precision for the different variables (see Annex 1-2, lower part!) at lab of SSAB EMEA, Borlänge, the following variables were recommended to be studied in a Round Robin of used mill emulsion: oil content, SAP, TAN, Ashes, Fe tot and pH.

Samples

Sample type 1: To each participant 1000 ml of emulsion from SSAB cold rolling mill was sampled during production 2009-11-23. The method for sampling is depicted in Annex 2.

Sample type 2: To each participant 125 ml of rolling oil concentrate from the batches that were represented in the sampled mill emulsion – the three last deliverances in three equal parts. The differences between these batches were insignificant.

Analysis

Analysis work at the participants laboratories was performed on 2009-11-26, three days after sampling. The recommended routine before start of lab work is described in Annex 3. Double samples were suggested.

Result presentation. Statistical evaluation

The results for each variable are shown on one and the same page in a table, in a graph and with a note on outlier tests Grubbs 1 and Grubbs 2. Also on same page at bottom are short outlines for the methods used at the different laboratories.

The straight line in the graph shows the mean value and the dotted lines the standard deviation (1s) after removal of outliers.

In cases when single results of pairs differ, they are indicated in the graph with a circle. If they are identical, or if only one sample have been analysed, a cross is denoted. A comment on single samples for each variable is written in the table with the result figures.

Although a low number of participants, as most 7 labs and 5 double results, for some variables, the mean value tests Grubbs 1 and Grubbs 2 are applied.

In Grubbs 1 (blue color in graph) the individual mean value is compared to the overall mean value of the population. In Grubbs 2 two mean values are tested at the same time (the two highest values, the two lowest values or the highest and the lowest value). An outlier indicates that the accuracy is not good enough.

The Grubbs test: G is used for a set of data in ascending order

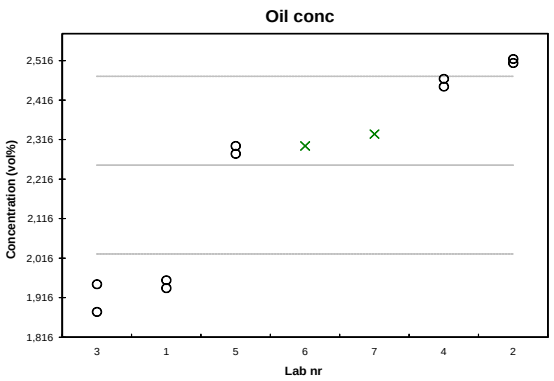
$$G = \left| \frac{g_p - \bar{g}}{s} \right|$$

g_p = extreme value $\bar{g}$ = mean value s = standard deviation

If G > a critical value according to a table value, the result value tested is rejected at 95% confidence level.

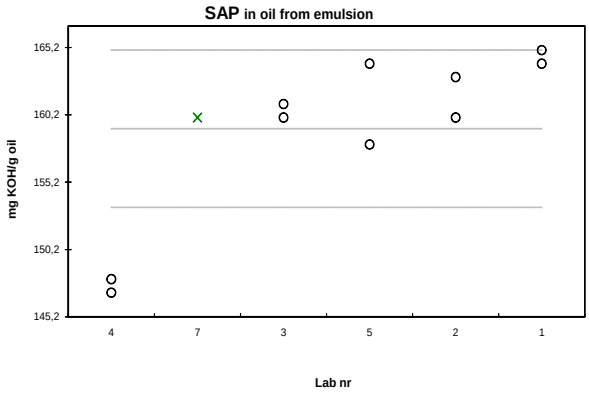
The statistical evaluation was made on software developed by Swerea KIMAB.

Results. Sample type 1



		Number of labs		7								
		Mean		2,25								
		Stdev		0,22	9.8%							
		No outlier acccording to Grubbs 1 and Grubbs 2										
		Lab nr		**Sample 1**	**Sample 2**							
		3		1,88	1,95							
		1		1,94	1,96							
		5		2,30	2,28							
		6		2,30	2,30	Just one sample analysed						
		7		2,33	2,33	"	"					
		4		2,45	2,47							
		2		2,51	2,52							

Lab nr	**Method**
3	Emulsion + PE-benzin and salts. 30 min shaking. Separation. Filtration glass-wool. 2:nd extr for settled water ph. Evap of org ph. Gravim.
1	Emuls + PE-benz+isoprop+salts. 30 sec man shak. Separation. Dispatch of water ph. Org ph filtr in 4 fold paper OOM. Evap. Gravim.
5	Rot evap of emul. Dissol in PE-benz. Filtr glass-wool. Evap. Gravim.
6	IR- balance. Sartorius MA 40
7	Rot evap of emul. Dissol in PE-benz. Filtr . Evap. Gravim.
4	IR- balance. Sartorius MA 40
2	IR- balance. Sartorius MA 30
Not:	Oil dens = 0.927 g/cm^3

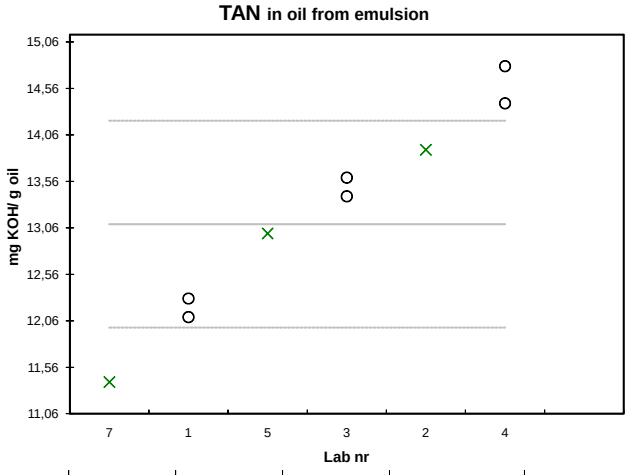


Number of labs	6	
Mean	159	
Stdev	5,8	3,6%

No outlier acccording to Grubbs 1 and Grubbs 2

Lab nr	Sample 1	Sample 2	
4	148	147	
7	160	160	Just one sample analysed
3	161	160	
5	164	158	
2	163	160	
1	165	164	

Lab nr	Method
4	Oil heated with 0,5 M KOH in EtOH. Titr with 0,5 M HCl. Fenolftalein+methylblue.
7	ISO 6293-1:1996 conf. DIN 51559 TI. Close to meth of lab 1 and 5
3	Oil diss in toluene+EtOH+indic. Add of surpl 0,5 ethanolic KOH. Boil 30 min. Titr 1 M HCl
5	Modified DIN 51559-1: 2009-04. Close to meth of lab 1 and 7
2	Oil diss in toluene50%,isoprop49%,deion w1%. Boiled with surplus KOH in EtOH. Titr 0,5 M HCl. Fenolftalein.
1	Oil diss in MEK. Boil 30 min in surpl 0,6 M KOH in EtOH. Titr 0,5 M HCl. Fenolftalein. Close to meth of lab 5 and 7

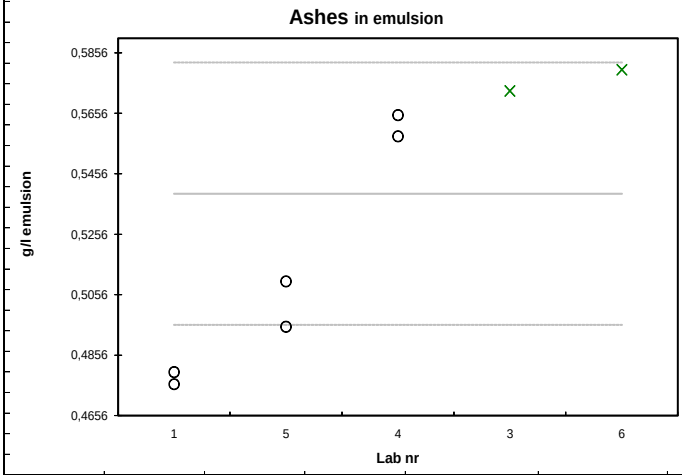


Number of labs	6	
Mean	13,1	
Stdev	1,1	8,4%

No outlier acccording to Grubbs 1 and Grubbs 2

Lab nr	Sample 1	Sample 2	
7	11,4	11,4	Just one sample analysed
1	12,1	12,3	
5	13	13	
3	13,4	13,6	
2	13,9	13,9	
4	14,5	14,8	

Lab nr	Method
7	DIN 51558 T2 (corr to ISO 6618:1997)
1	Oil diss in toulene50%+isoprop49%+deion w 1%. Titr 0,1 M KOH in isoprop. Fenolftalein. (ISO 6618:1997)
5	Modified DIN 51558-1:1979-07
3	As lab nr 1. Also labs 2, 5 and 7 very close.
2	As lab nr 1.
4	Oil diss in isoprop. Titr 0,1 M KOH in isoprop. Fenolftalein.

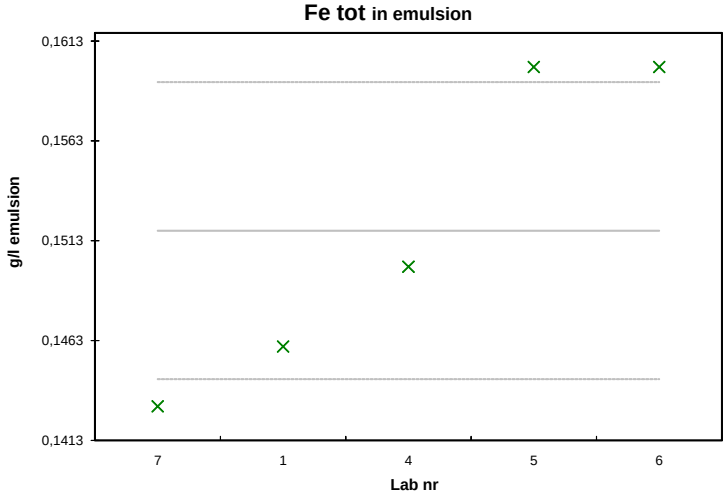


Number of labs	5
Mean	0,539
Stdev	0,043
	8,0%

No outlier acccording to Grubbs 1 and Grubbs 2

Lab nr	Sample 1	Sample 2	
1	0,476	0,480	
5	0,510	0,495	
4	0,565	0,558	
3	0,573	0,573	
6	0,58	0,58	Just one sample analysed

Lab nr	Method
1	Water in emulsion evap in Pt-cruc on hot plate. Burned in Thermolyne furn 775˚C, 30 min.
5	Water in emulsion evap in Pt-cruc on hot plate. Burned in furn 800˚C. 30 min
4	Water in emulsion evap in Pt-cruc on sand compress. Burned in bunsen flame. Then furn 775˚ C. 30 min.
3	Drying at 105˚C 24 hrs. Furn 775˚C 60 min.
6	Water in emulsion evap in Pt-cruc on hot plate. Furn 700˚C.

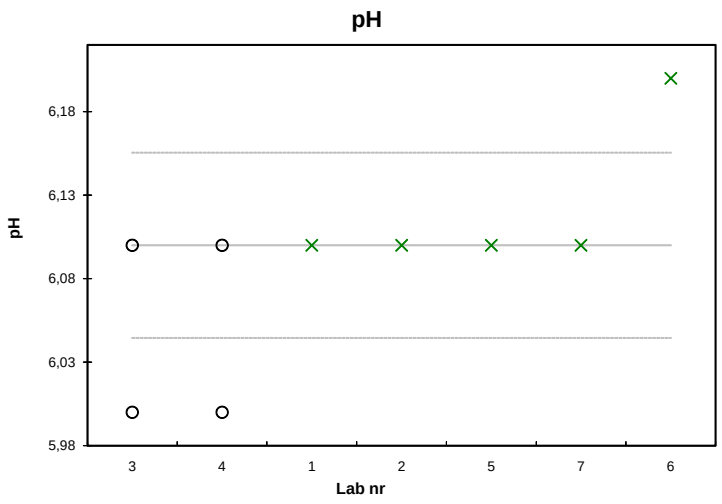


Number of labs	5	
Mean	0,152	
Stdev	0,007	4,6%

No outlier acccording to Grubbs 1 and Grubbs 2

Lab nr	Sample 1	Sample 2	
7	0,143	0,143	Just one sample analysed
1	0,146	0,146	
4	0,150	0,150	
5	0,16	0,16	
6	0,16	0,16	Just one sample analysed

Lab nr	Method
7	ICP-OES after HCl treatment
1	AAS after dissolution of ashes in HCl. Boiled.(some HF for SiO_2 a o)
4	AAS. Water in emulsion boiled with H_2SO_4
5	ICP. Emulsion in HNO_3. 200 bar.
6	ICP. Emulsion boiled with HCl



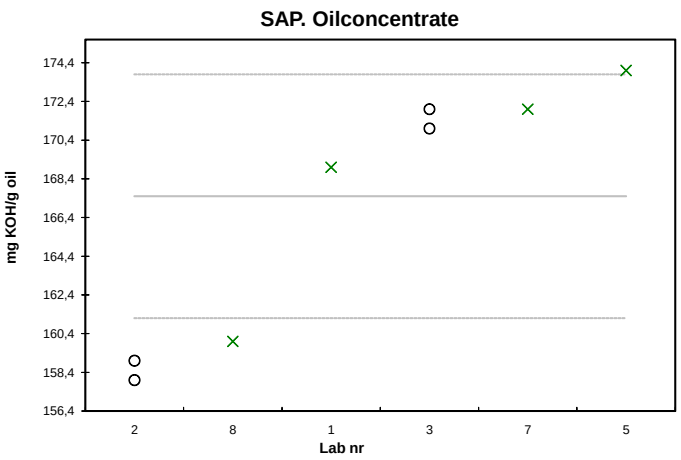
Number of labs	7	
Mean	6,1	
Stdev	0,06	1,0%

No outlier acccording to Grubbs 1 and Grubbs 2

Lab nr	Sample 1	Sample 2		
3	6,1	6,0		
4	6,0	6,1		
1	6,1	6,1		
2	6,1	6,1		
5	6,1	6,1		
7	6,1	6,1	Just one sample measured	
6	6,2	6,2	"	"

Lab nr	Method
3	Metrohm 740, combined electrode. Emulsion 25˚C +/- 1˚C
4	Mettler DL 25. Comb electr DG 111-SC. Emulsion 25˚C +/- 0,5˚C
1	Metrohm 740, combined electrode. Emulsion 25˚C +/- 1˚C
2	WTW pH 340i. DIN 51369 7/81
5	WTW. 20˚C
7	pH Meter
6	

Results. Sample type 2

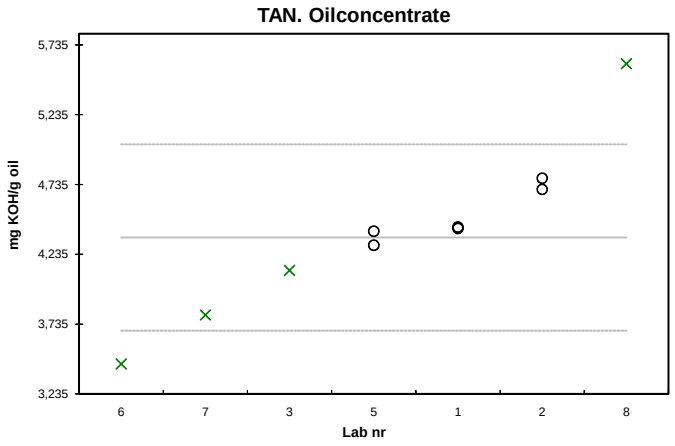


Number of labs	6
Mean	168
Stdev	6,3
	3,8%

No outlier acccording to Grubbs 1 and Grubbs 2

Lab nr	Sample 1	Sample 2	
2	159	158	
8	160	160	Just one sample analysed
1	169	169	
3	172	171	
7	172	172	Just one sample analysed
5	174	174	

Lab nr	Method
2	Oil diss in toluene50%,isoprop49%,deion w1%. Boiled with surplus KOH in EtOH. Titr 0,5 M HCl. Fenolftalein.
8	Oil boiled 60 min with 0,5 M KOH in EtOH. Rinsed with isoprop.Titr with 0,5 M HCl. Fenolftalein.
1	Oil diss in MEK. Boil 30 min in surpl 0,6 M KOH in EtOH. Rinsed with EtOH.Titr 0,5 M HCl. Fenolftalein.
3	Oil diss in toluene+EtOH+indic. Add of surpl 0,5 ethanolic KOH. Boil 30 min. Titr 1 M HCl
7	ISO 6293-1:1996 conf. DIN 51559 TI. Close to meth of lab 1 and 5
5	Modified DIN 51559-1: 2009-04. Close to meth of lab 1 and 7

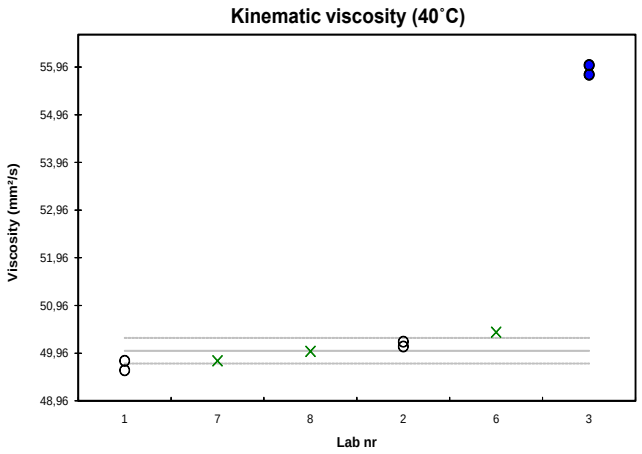


Number of labs	7
Mean	4,36
Stdev	0,67 15%

No outlier acccording to Grubbs 1 and Grubbs 2

Lab nr	Sample 1	Sample 2			
6	3,45	3,45	Just one sample analysed		
7	3,8	3,8	"	"	"
3	4,12	4,12			
5	4,3	4,4			
1	4,42	4,43			
2	4,78	4,70			
8	5,6	5,6	Just one sample analysed		

Lab nr	Method
6	Titr with 0,1 M KOH
7	DIN 51558 T2 (corr to ISO 6618:1997)
3	As lab nr 1. Also labs 2, 5 and 7 very close.
5	Modified DIN 51558-1:1979-07
1	Oil diss in toulene50%+isoprop49%+deion w 1%. Titr 0,1 M KOH in isoprop. Fenolftalein. (ISO 6618:1997)
2	As lab nr 1.
8	Oil diss in isoprop. Heat to boiling. Titr immediately with 0,1 M NaOH. Fenolftalein



		Number of labs		5				Number of labs		6					
		Mean		50				Mean		51					
		Stdev		0,27	0,54%			Stdev		2,3					
		Outlier acc to Grubbs 1 excluded						Outlier included							
		Lab nr		**Sample 1**	**Sample 2**										
		1		49,8	49,6										
		7		49,8	49,8	Just one sample measured									
		8		50,0	50,0	"									
		2		50,2	50,1										
		6		50,4	50,4	Just one sample measured									
		3		55,8	56,0										

Lab nr	**Method**
1	Ubbelohde-Viscosimeter. Schott Typ 525 20 / II. SS-ISO 3105
7	Micro Viscosimeter AMV Fa. Paar
8	
2	Stabinger Viscosimeter SVM 3000. ASTM 7042 7/04
6	Ubbelohde kapillär
3	

Comments

Sample type 1, used mill emulsion:

Oil content

The two labs with the lowest results (nr 3 and 1) both start with solvent extraction of emulsion, without previous evaporation of water from emulsion. The other laboratories either use IR-balance or start their solvent extraction procedure by evaporating water from the emulsion. Lab 5 and 7 have almost identical methods, so are their results very close. A little more surprising is that results from IR-balance-method differ a lot, from 2,30 to 2,52 vol%.

SAP

In "statistical terms" no outlier was identified, but in "chemical terms" there was one. This result may be explained by a difference in analysis method. This "outlier", lab 4, dissolves first the oil in EtOH and the added surplus base, in contrary to the other participants who add MEK or a mixture of solvents with Toluene for a dissolution of oil to start with, before adding of KOH in EtOH in surplus prior to boiling.

TAN

There were no overlapping result values between labs for this variable. The result pairs presented were quite close for each participant. The highest TAN was given from a laboratory with a deviating method. Lab 4 dissolves the oil in isopropanol (2-Propanol), while other labs use a mixture of solvents (Toluene50%, 2-Propanol49%, deion. Water 1%).

Ashes

The performance of the two main operations in the methods concerned differed to some extent. Distribution of results can to the mayor part be explained by these differences. The lowest values of ash content emanate from two labs (nr 1 and 5) with quite brutal methods: evaporation in Pt-crucible on hot plate and burning at

high temperature, 775˚C and 800˚C, in furnace. The higher results were received after lower temperatures and longer times.

Fe tot

From the brief information regarding methods just one comment is given. The highest values come from two participants using acid treatment directly on emulsion prior to ICP.

pH

Minor differences.

Sample type 2, fresh oil concentrate

SAP

The low value of lab 8 (external participant) can be related to a somewhat deviating method; the oil is boiled with ethanolic KOH without prior dissolution in a little less polar solvents as MEK or Toluene-Isopropanol. No obvious explanation from the method information accessible to the low value for lab 2. Other results were quite similar.

TAN

Also here lab 8 is a “chemical outlier” due to a differing method. Isopropanol is used instead of mixture containing Toluene at other labs.

Kinematic viscosity (40˚C)

At least three different brands of viscosimeters give very close results for 5 of the six laboratories. As for the statistical outlier we have no information to share.

General comment

Compared to the measurement uncertainty at SSAB the results of this proficiency testing were fairly good (Annex 1-2). It is also interesting to see the impact of differences in methods. With these routines for the realization of this testing, it seems possible to compare used cold rolling mill emulsion of this type.

Participants

Marie Ekman, Ph.D
Manager, Chemical and Environmental Analysis

Göran Lindén M Sc. Chemist

Sapa Technology
SE-612 81 Finspong, Sweden

Dipl.-Ing. Tilo Reichardt

Abteilungsleiter Galvanotechnik und Oberflächenveredelung/Head of Department Plating and Surface Finishing
Fachbereich Stahlveredelung und -anwendung/Division Steel Finishing and ApplicationVDEh-Betriebsforschungsinstitut GmbH, Sohnstr. 65

DE-40237 Düsseldorf

Teemu Yli-Kovero
Laboratory Manager, Ruukki Metals

Raija Lintala

Rautaruukki Oyj
Harvialantie 420
FI-13300 Hämeenlinna

Dr.
Frank Kleppe

Chemisches Labor
Salzgitter Flachstahl GmbH
Eisenhüttenstraße 99

DE-38239 Salzgitter

Dr.Hubert Duchaczek
Umwelt- und Betriebsanalytik (B4P4)

Heinz Gsaller

Fachtechniker Umwelt- und Betriebsanalytik.
B4 Technischer Service und Energie
Prüftechnik und Analytik B4P

Voestalpine Stahl GmbH

Voestalpine-Straße 3

AT-4020 Linz

Dr. Martin Raulf

TKS

Werkstoffkompetenzzentrum
Team Oberflächenanalytik
Eberhardstr. 12

DE-44145 Dortmund

Anders Janols

Chemical laboratory

SSAB EMEA, Borlänge

SE-78184 Borlänge

(Coordinating laboratory)

Study on some properties important for a Round Robin of a mill emulsion

Scope

To test the physical and chemical stability of SSAB:s present cold rolling mill emulsion, regarding some commonly investigated variables, and its suitability for Round Robin.

Emulsion

An ester based, physically quite stable emulsion with an oil content of about 2,5 %vol. Sampled from a system at a mill during rolling

Method

For sampling method: see annex 2! The prolonged period between sampling from mill of a big volume and distributing the emulsion into small bottles for analysis was due to the intention of getting a cleaner and more stable emulsion with some reduced oil content and smaller particles.

For storing and preparation methods in accordance with suggested plan for RR: see annex 3!

For analysis methods: see annex 4!

Double samples were analysed at every occasion

Result

See annexes 1-2 and 1-3!

Conclusion

After comparing the result with the documented precision for the different variables at lab of SSAB EMEA, Borlänge (annex 1-2!) we recommend the following variables to be studied in a Round Robin of mill emulsion: oil content, SAP, TAN, Ashes, Fe tot and pH.

AJO / 2009-08-10

Method test for Round Robin of used cold rolling emulsion[1/]

Annex 1-2

Sampling 2009-05-26 kl 08.19 at SSAB cold rolling mill to 25 liter carboy with tap

Sampling from bottom[2/] (after 3 hrs spontaneous separation in carboy, upside down!) to bottles 2009-05-26 kl 11.25

Start analysis	Day , sample	Lab data nr	Particles µm	Oil cont %vol	SAP mg KOH/g	AN mg KOH/g	Ashes g/l	Iron tot g/l	pH	Cond µS/cm x0,01	Not
2009-05-26 11.55	0A	09-642,1	1,50	2,16	162	14,5	0,640	0,208	5,7	3,22	
"	OB	09-642,2	1,49	2,19	160	14,6	0,648	0,216	5,7	3,22	
(Night)											refrig 8˚C
2009-05-27 08.40	1A	09-642,3	1,45	2,15	162	14,7	0,648	0,227	5,7	3,21	1A,B 40˚C
"	1B	09-642,4	1,46	2,18	162	14,8	0,644	0,224	5,7	3,21	"
(Nights)	*(In days sample for later analysis were stored at room temp)*										refrig 8˚C
2009-05-29 08.40	3A	09-642,5	1,43	2,17	158	14,7	0,604	0,235	5,7	3,36	3A,B 40˚C
"	3B	09-642,6	1,38	2,18	158	14,9	0,608	0,224	5,7	3,38	"
(Week-end)											amb (23˚C)
2009-06-01 08.40	6A	09-642,7	1,41	2,16	161	14,7	0,628	0,241	5,9	3,39	6A,B 40˚C
"	6B	09-642,8	1,40	2,16	161	14,8	0,620	0,239	5,8	3,37	"

Documented precision att SSAB chem lab, 2s (%)	8,8	3,0	1,2	6,4	6,4	11	0,4	3,0
Within 2s in this test?	Yes, but evident trend	Yes	Yes	Yes	Yes	Not day 6	Not day 6	Not day 3 and 6

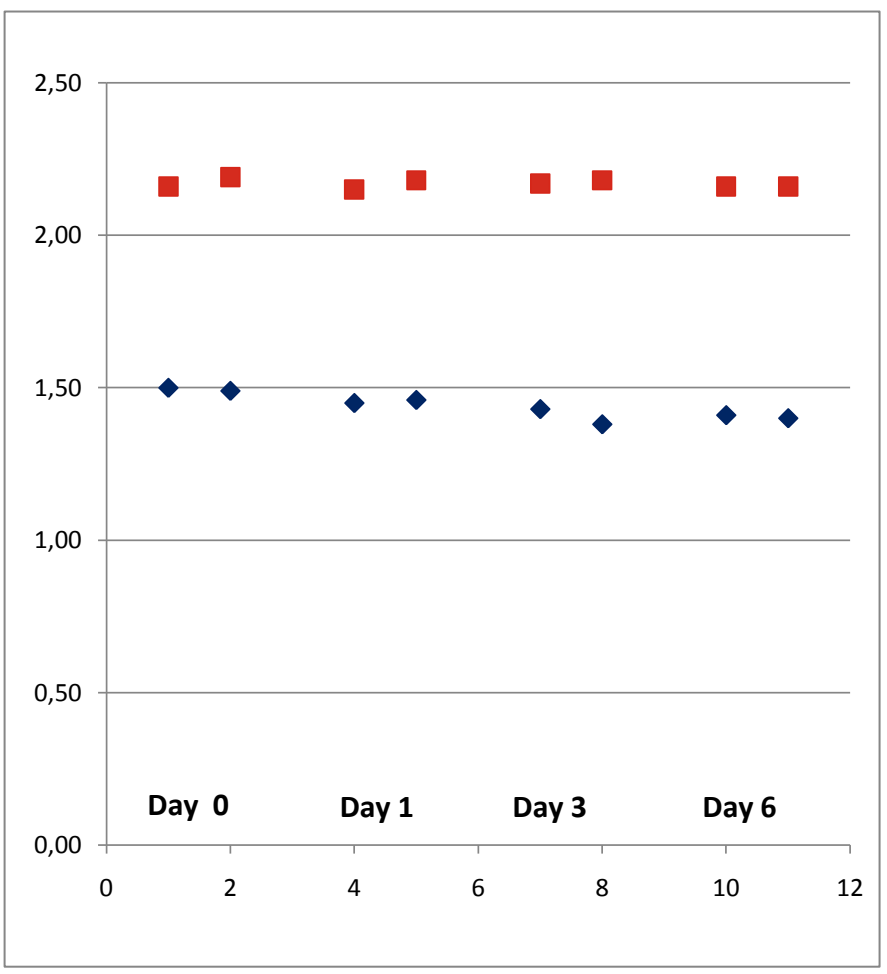
[1/]= the results for some of the variables are presented with one more digit than is significant for SSAB:s result

[2/]= first into a 10 liter bottle for repeated turning around and randomly distribution of emulsion in 1 liter sample bottles for Round Robin

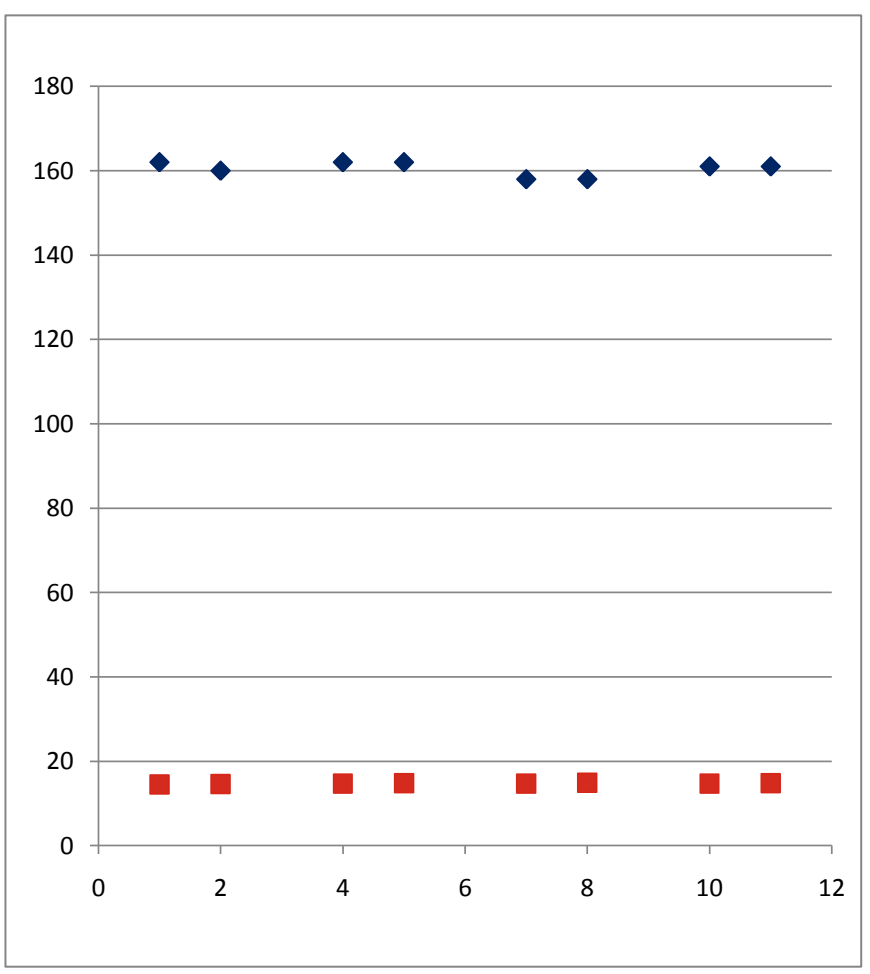
Method test for Round Robin of used cold rolling emulsion

Annex 1-3

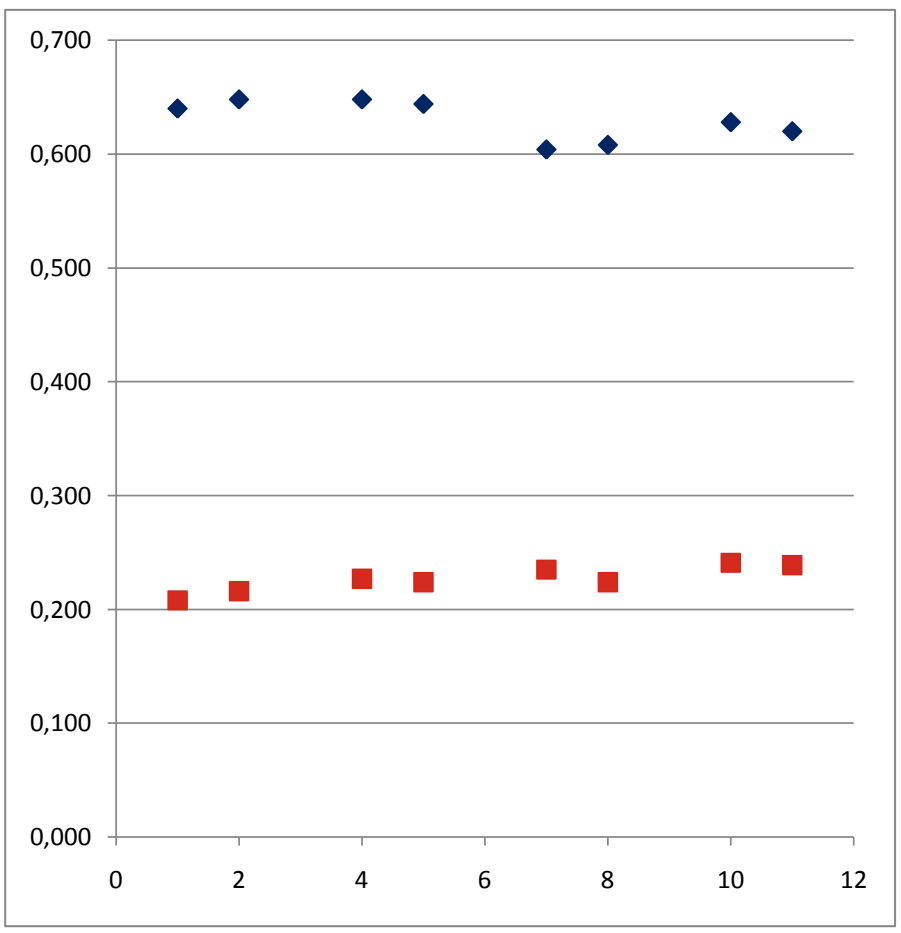
Oilcont(quadr). Partsize, median (rhombi)



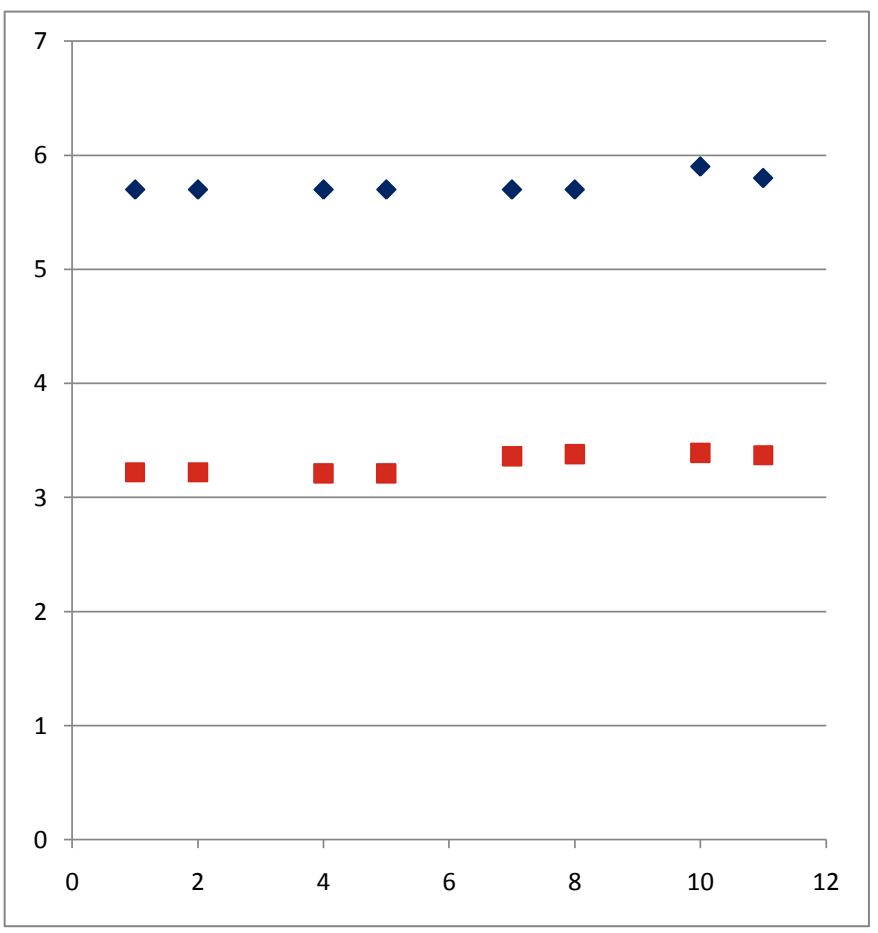
SAP (rhombi). TAN (quadr)



Ashes (rhombi). Iron tot (quadr)



pH (rhombi). cond x 0,01 µS/cm (quadr)



2009-06-02/ AJO

Annex 2

Method for sampling of mill emulsion for Round Robin

- Fill emulsion, from mill system during rolling, into a 25 liter carboy (PE-HD) with tap (95% of vol)
- Turn it upside-down (screw cap with tap downwards)
- Keep the vessel unmoved in the same position for 3 hrs
- Cut a hole, 20 mm diameter, in the upper surface (bottom!) of the carboy
- Open the tap, the vessel still unmoved, and fill a 10 liter bottle (screw cap, PE-HD)
- Distribute emulsion randomly, from the 10 liter bottle, in small portions into 1 liter bottles (screw cap HDPE) for distribution to Round Robin

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Annex 3

Round Robin. Cold rolling emulsion from mill.

Transport and lab work

- The samples are sent from co-ordinating lab in afternoon the day of sampling from mill (day 0)
- A mode of conveyance is booked that guarantees arrival to participants lab before day 3. That is sampling on Mon (day 0), start analyse work on Thu (day 3)
- After arrival to lab, the sample is stored at room temperature before start of work with samples
- All participating laboratories perform their analyse on day 3
- Before start of work, bottle with sample should be heated to 40˚C in a water-bath and then vigorously shaken before sampling for analysis
- Double samples should be analysed
- **Participants are free to use methods of their own choise.** Outlines of the methods used should be enclosed to the result (Recommended level of details as in annex 4)
- Results and info on used methods as soon as possible to anders.janols@ssab.com

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Annex 4

Methods for the tested variables at chem lab, SSAB EMEA, Borlänge

Particle size

20-30 minutes after sampling or re-warming (40˚C) and shaking. Turning gently twice just before taking out test portions for measuring in Malvern Mastersizer Micro+. Presentation: Standard 5 OHD (1,5295 0,1000 in 1,3300)

Oil content

Internal gravimetric method developed in cooperation with Akzo Nobel and Quaker Chemicals. Solvent extraction (Petroleum spirit 60-80˚C, Propan-2-ol, Sodium chloride/10 w/w%), filtration of organic phase (two four-folded OOM paper), evaporation.

SAP

SS-ISO 6293-1 (ASTM D 94). Oil dissolved in Ethyl Methyl ketone, heated (boiling) with surplus KOH in ethanol and titrated with 0,5 M HCL. Fenolftalein as indicator.

TAN

Modified ASTM D 974. Oil dissolved in mixture of solvents (Toluene 50%, Propan-2-ol 49%, deion water 1%). Titrated with 0,1 M KOH in Propan-2-ol. Fenolftalein.

Ashes

Water in emulsion evaporated in Pt-crucible on hot plate. Burned to ashes in a Thermolyne 48000 furnace. 775˚C, 30 min.

Fe tot

Ash dissolved in konc HCl (37%). AAS.

pH

pH-meter Metrohm 744, combined electrode. Emulsion 25˚C +/- 1˚.

Conductivity

Schott CG 853 P. Measuring cell WTW Tetralon 325 (with temp sensor). Emulsion 25˚C +/- 1˚.

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JERNKONTORET

Box 1721, SE-111 87 Stockholm, Sweden Kungstr dg rdsgatan 10
Telephone +46 8 679 17 00 Fax +46 8 611 20 89
E-mail office@jernkontoret.se www.jernkontoret.se