

Managing total reduced sulfur emissions at a Canadian kraft pulp mill

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ABSTRACT: This study addresses the challenges a dissolving-grade pulp mill in Canada faced in 2014 in meeting its total reduced sulfur (TRS) gas emission limit. These emissions from the recovery boiler exit are controlled by passing the boiler exit gas through a TRS scrubber system. The mill employs a cyclonic direct contact evaporator to concentrate black liquor to firing solids content. The off-gases from the direct contact evaporator flow to the effluent gas control system that consists of a venturi scrubber, a packed bed scrubber, and a heat recovery unit. Emissions of TRS greater than the regulated limit of 15 ppm were observed for a 4-month period in 2014. The level of emissions measured during this period was significantly higher than about 12 ppm, the expected average value based on historic experience. The problem persisted from mid-June 2014 until the annual mill shutdown in October 2014.

The main TRS components detected and the performance of the Teller scrubber in capturing them are examined. Other potential causes for these emissions are identified, including mechanical problems such as broken packing in the TRS packed bed scrubber, broken baffle plates in the scrubber, and cyclone evaporator leaks causing air ingress. Repairs were carried out during the mill shutdown, which eliminated the TRS emissions problem.

Application: Mills can use this information as a guide for identifying and removing sources of TRS emissions from alkaline pulp mill recovery systems.

A mill located in Canada produces dissolving-grade pulp. The mill pulps northern hardwoods, particularly, maples and poplars, in batch digesters. The cooking process for the viscose pulp production uses continuous batch cooking, terminated by blowing the digesters. Two blow tanks receive cooked pulp from the digesters. The blow tanks are vented to a blow heat recovery and low volume, high concentration noncondensable gas (LVHC-NGC) total reduced sulfur (TRS) collection system. The chemical recovery plant operates a seven-body, five-effect evaporator train. Evaporator NCG is collected and sent to the LVHC-NGC collection system where, together with digester NCGs, it is burned in a power boiler fueled by oil and bark. The mill's recovery boiler emission is subject to the pulp and paper industry emission regulations under the provincial Clean Air Act, which for kraft mills regulates a maximum allowable emission of 15 ppm of sulfur on a 4-hour rolling average.

The characteristics of the four TRS compounds dealt with in this study are shown in **Table I** [1,2].

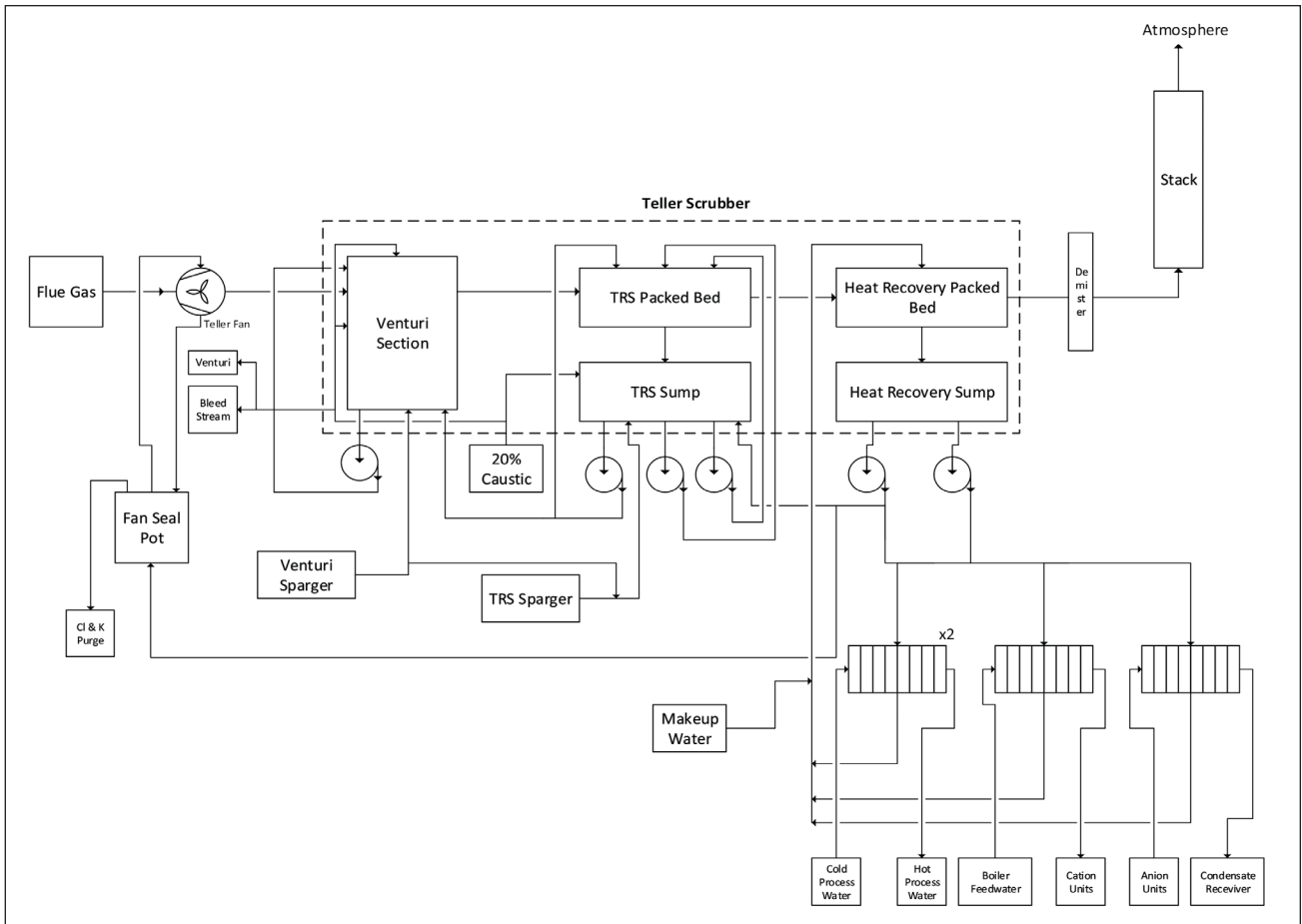
THE PROBLEM

An increase in TRS emissions from the mill over a period of weeks or months could result from several causes. One is a substantial increase in TRS generation within the pulping and recovery areas of the mill, which could overload the TRS capture, collection, and destruction capabilities of the Teller system. Another is failure of the sulfur capture processes to per-

form as expected. A third is mechanical failure that causes release of TRS-containing gases to the environment. The possible underlying causes are many and complicated. A plan was developed by the mill's technical staff to identify the sources of TRS emissions. This paper reviews the background and results from the technical team's investigation and findings.

TRS Compound	Characteristic Odor	Odor Threshold, ppb	Explosive Limits, %	
			Lower	Upper
Hydrogen sulfide (H ₂ S)	Rotten eggs	0.4–5.0	4.3	46
Methyl mercaptan (CH ₃ SH; MM)	Rotten cabbage	0.6–3.0	3.9	22
Dimethyl sulfide (CH ₃ SCH ₃ ; DMS)	Vegetable sulfide	0.3–3.0	2.2	9
Dimethyl disulfide (CH ₃ S ₂ CH ₃ ; DMDS)	Decayed vegetables	15.5	-	-

I. Characteristics of total reduced sulfur (TRS) gases generated in kraft pulp mills.



1. Teller scrubber process flow diagram.

Factors influencing TRS emissions at the mill

The amount of TRS emissions from alkaline scrubber systems such as the patented Teller scrubber (Teller Environmental Systems; Worchester, MA, USA) [3] at the mill depends on several factors. The study asked the following questions related to these factors: (1) Is the TRS scrubber being overloaded by the total amount of TRS entering it; (2) is the TRS scrubber simply not capable of removing some of the TRS compounds sent to it (organosulfur compounds are of particular concern here); and (3) has the performance of the scrubber been degraded with time through failure of its internal components?

Each factor was evaluated by first reviewing the chemical processes involved in TRS generation and its capture, then evaluating the TRS scrubber performance using data from the mill, and, finally, examining the mechanical factors that could have an impact on TRS scrubber performance.

Scrubber system

The mill uses a Teller TRS scrubber system that employs a wet scrubber [3]. The main purpose of the scrubber system is to remove TRS and fine particulates from the recovery boiler flue gases to comply with the air pollutants emission limit.

Flue gases from the recovery boiler pass through cyclone

evaporators, an electrostatic precipitator, and the TRS scrubber before being released through the stack to the atmosphere. Dusts and fine particles in the flue gases are removed in the electrostatic precipitator, prior to the gases entering into the scrubber. The scrubber system consists of three different sections, namely the venturi section, the TRS section, and the heat recovery section. **Figure 1** is a schematic diagram of the mill's Teller scrubber system.

In the venturi section, particulates larger than about 1 micron in diameter are removed through inertial impaction with the scrubbing liquor. The liquor is atomized by shear as it contacts the contaminated gas in the throat of the venturi [4]. The fine droplets of the atomized scrubbing liquor absorb TRS gases. A small bleed stream off the venturi sump, containing mostly sodium sulfate and sodium carbonate (Na_2CO_3), is recycled to the cyclone evaporators. The pH of the scrubbing liquid in the sump is maintained at or above 9 by addition of make-up caustic solution of strength 267 g/L.

The main component of the scrubber system is the TRS absorption bed. It utilizes a packed bed scrubber that consists of a chamber containing layers of multiple shaped packing materials such as tellerets or Berl saddles that provide a large surface area for liquid-gas contact [5]. The TRS section employs a caustic solution to remove hydrogen sulfide (H_2S)

and methyl mercaptan (CH_3SH ; MM). It is recirculated from the TRS sump to the top of the bed and sprayed onto the packing at three different locations where it reacts with the TRS gases and carbon dioxide (CO_2). It also serves to remove particulate matter that has been deposited on the packing spines. The three spray streams each have a flow rate of 3000 gal/min. The high liquid-to-gas flow ratio enhances gas absorption. The pH of the scrubbing liquid in the sump is maintained at or above 8.8 by addition of a makeup caustic solution of strength 267 g/L.

The scrubbed flue gases then move to the next section called the heat recovery bed. Here, heat is extracted from the gases for heating boiler feed water and filtered water used in the bleaching and stock preparation areas of the mill. No TRS removal occurs in this bed.

Hypochlorite is used on the Teller stack before the flue gases exit to atmosphere when needed by the mill to meet TRS emissions limits. Hypochlorite is an effective and straightforward way to oxidize dimethyl sulfide (CH_3SCH_3 ; DMS) and dimethyl disulfide ($\text{CH}_3\text{S}_2\text{CH}_3$; DMDS). Hypochlorite can also oxidize H_2S .

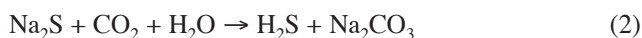
CHARACTERISTICS OF TRS EMISSIONS

At this mill, major TRS gases going to the Teller scrubber are released during the burning of black liquor in the recovery boiler and concentration of black liquor in direct contact evaporators, and also from the smelt dissolving tank. The TRS components include H_2S , MM, DMS, and DMDS.

Hydrogen sulfide

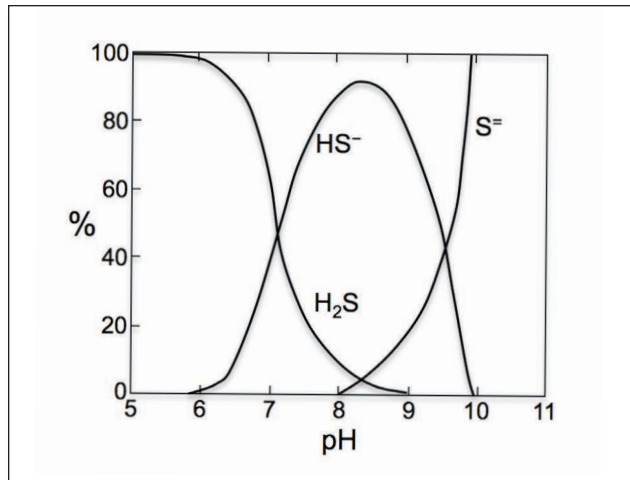
Hydrogen sulfide is released during black liquor combustion in the recovery boiler furnace, in the smelt dissolving tank, and in direct contact (cyclone) evaporators. In the recovery boiler furnace, H_2S emissions occur because of low char bed temperature and incomplete oxidation of the lower furnace gases caused by inadequate mixing of secondary and tertiary air. Poor air distribution in the recovery furnace has been demonstrated by high carbon monoxide (CO) (>200 ppm) values in flue gases at the recovery stack exit as reported in previous studies [1].

Smelt is another source of H_2S emission, where H_2S can be produced by the reaction of sodium sulfide (Na_2S) in the smelt with water vapor and carbon dioxide (CO_2) [6], as in Eq. (1) and Eq. (2):



In a direct contact cyclonic evaporator, CO_2 in the flue gas displaces H_2S from black liquor. Emission of H_2S from black liquor is favored by high sulfide content and low pH (<12).

Hydrogen sulfide is an acidic gas that ionizes in alkaline solutions; it is not volatile in its ionized forms. Its volatility is determined by the distribution of its three solution-phase species,



2. Distribution of aqueous hydrogen sulfide species over the pH range 5–11 at 25°C.

ies, H_2S (aq), HS^- , and S^{2-} , and how these species shift to the left as process conditions change, according to Eq. (3):

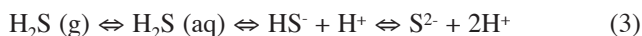


Figure 2 shows the distribution of aqueous hydrogen sulfide species over the pH range of 5–11 at 25°C. HS^- , the hydrosulfide ion, is the predominant form in aqueous solutions at pH <7 to pH <9.5. Alkaline scrubbing is normally done at pH 9.0–9.5, using caustic to form sodium hydrosulfide (NaHS). Carbon dioxide is coabsorbed when H_2S is scrubbed from flue gas. It reacts with sodium hydroxide (NaOH) to form primarily sodium bicarbonate (NaHCO_3) and smaller amounts of sodium carbonate (Na_2CO_3) [7].

The absorption of CO_2 into caustic solution is a relatively slow process compared to H_2S absorption, but some is always absorbed. In a previous study where the gases contained 1% H_2S and 10% CO_2 , about 5%–7% of the CO_2 was scrubbed and the ratio of $\text{H}_2\text{S}/\text{CO}_2$ removed was about 1.6 [7]. The ratio would be lower for systems such as this mill, where H_2S concentrations are much lower and CO_2 concentrations are higher in flue gas entering the Teller scrubber.

Methyl mercaptan

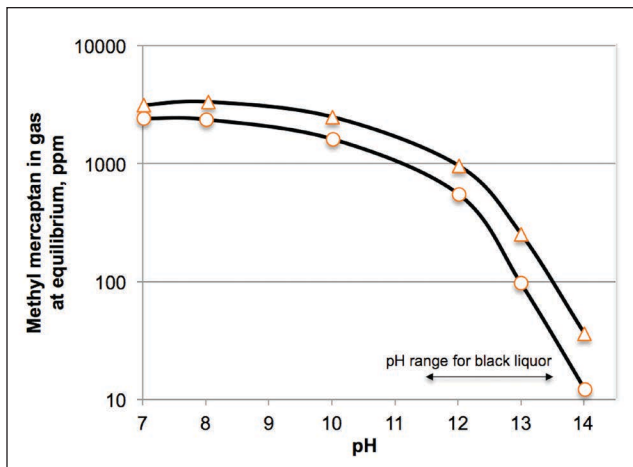
During pyrolysis, MM can be formed through the reaction in Eq. (4):



Because of its low solubility in black liquor, MM is readily stripped out in the cyclones. It can be formed in cyclone evaporators by the reaction in Eq. (5):



Mercaptan formation in black liquor increases with decreasing pulp kappa number. This means that digesters oper-



3. Concentration of methyl mercaptan in gases in contact with aqueous alkaline solutions for pH 7–14 at 80°C and 100°C.

ated with higher H factors can generate more mercaptan. Methyl mercaptan is a weakly acidic gas that ionizes to the mercaptide ion in alkaline solutions (Eq. [2]), and can be partially scrubbed by caustic solutions, as in Eq. (6):



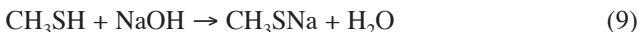
Figure 3 shows the equilibrium concentration of MM in the gas phase at different solution pH and two temperatures. These results are for a total concentration of 0.01 mole/L MM. As shown, high pH values prevent the release of mercaptan in the gas phase; therefore, less MM vapor is produced as the pH of black liquor increases.

Methyl mercaptan can be oxidized to DMDS via the reaction in Eq. (7). This reaction helps to reduce MM emissions, but it creates the more volatile DMDS.

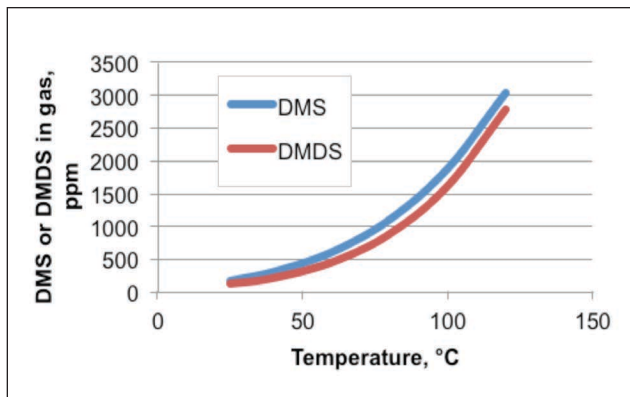


Dimethyl sulfide and dimethyl disulfide

These compounds, DMS and DMDS, can be emitted from many points within a kraft pulp mill, but the main sources are digester/blow tank systems and the direct contact evaporators. Most of the organosulfur TRS compounds are stripped from black liquor during evaporation and only a small fraction remains in the black liquor reaching cyclones. These gases are formed by the reactions in Eqs. (8–11):



Dimethyl sulfide and DMDS do not ionize in caustic solutions.



4. Concentrations of dimethyl sulfide (DMS) and dimethyl disulfide (DMDS) in gas in equilibrium with aqueous solutions containing 0.0001 mole/L of either sulfur compound.

They have limited solubility in water (DMS: 2% w/v at 25°C; DMDS: 2.5 g/L at 20°C) and even less solubility in salt solutions because of the salting-out effect [9]. They are very volatile and their solubility decreases with increasing temperature. **Figure 4** shows the remarkably high concentration of each TRS gas in equilibrium with an aqueous solution containing 0.0001 mole/L of the TRS compounds. Values in Fig. 4 were generated by using Henry's law, with constants from Sander [8].

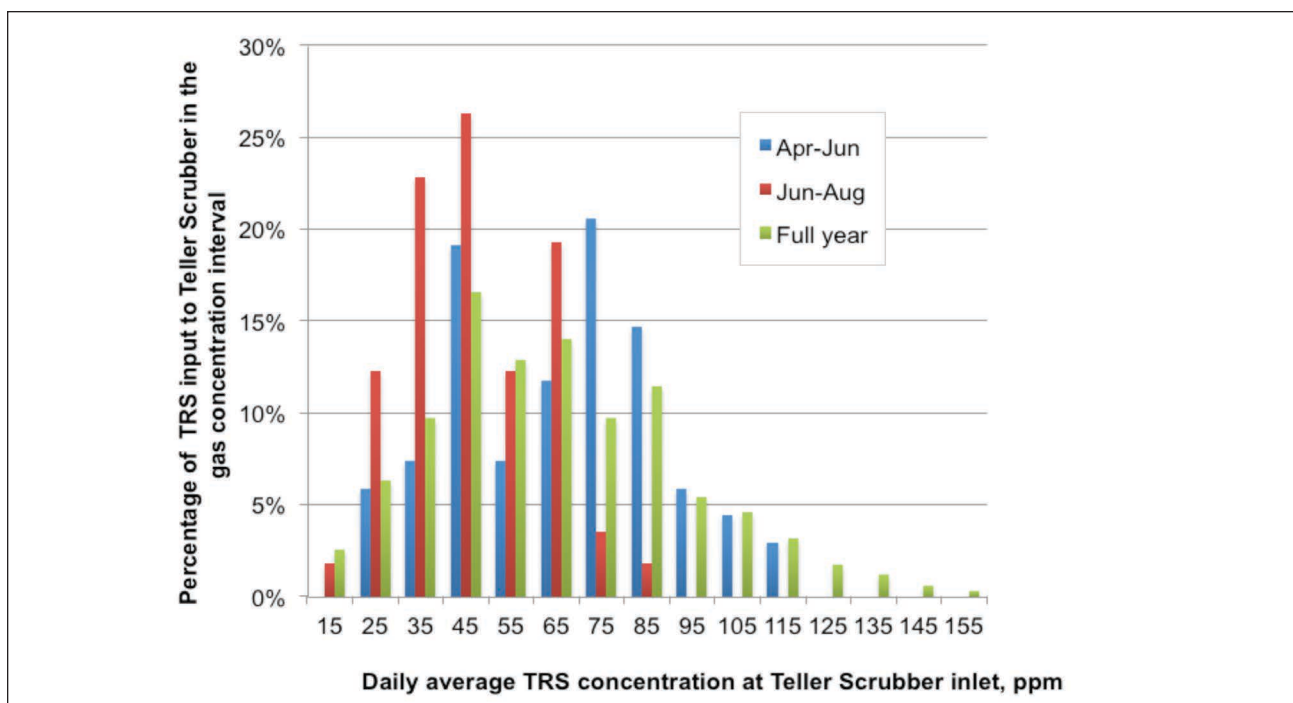
Dimethyl sulfide and DMDS are not effectively removed with a caustic scrubber. Reducing their emissions from the Teller system requires either (a) their removal upstream of the Teller system by, for example, increasing digester venting or (b) oxidation within the Teller system with hypochlorite as is practiced when needed by the mill [9].

TRS CAPTURE AND DESTRUCTION

TRS concentration at the scrubber inlet

The total concentration of TRS entering the scrubber system is continuously measured online by using an STI Model 43C TRS analyzer (Thermo Environmental Instruments Inc.; Franklin, MA, USA). **Figure 5** shows the distributions of TRS concentrations entering the Teller scrubber in terms of daily average TRS concentration. The data shown are based on daily averages for data collected over a 1-year period, August 12, 2013–August 12, 2014. Data for two subsets of the 1-year measurements are also included. They correspond to a 2-month period when TRS emissions were within the regulatory limits (designated as April–June 2014) and to a second 2-month period when the TRS emissions exceeded the regulatory limits (designated as June–August 2014). The distributions are similar, although the June–August 2014 distribution lies to the left, at lower TRS concentrations than the other two distributions. This means that the TRS concentrations entering the TRS scrubber were slightly lower during the period when TRS emissions were higher.

The average TRS concentrations and the standard deviations for each are shown in **Table II**. The average TRS entering the Teller scrubber is 11% higher during April–June 2014, the 2-month period when the TRS emissions were in compli-



5. Distribution of TRS concentrations entering the Teller scrubber in terms of daily average TRS concentration. Data shown are for a 1-year period (August 12, 2013–August 12, 2014), and for two 2-month subsets of the 1-year measurements.

ance. Further analysis showed that these two averages for the April–June 2014 and June–August 2014 periods are not statistically different, with a confidence limit of 99%.

It was concluded that differences in TRS input to the Teller system were not responsible for the increased TRS emissions from the scrubber during the mid-June–mid-October 2014 period.

Analysis Period	TRS (ppm)	
	Average	Standard Deviation
April 15–June 13, 2014	61.9	25.1
June 14–August 12, 2014	55.3	23.7
August 12, 2013–August 12, 2014	63.4	28.0

II. TRS emissions as daily averages and standard deviations for the three periods studied.

Sample Source	pH	Sulfate (SO ₄ ²⁻), g/L	Thiosulfate (S ₂ O ₃ ²⁻), g/L	Sodium Sulfide, g/L as Na ₂ O	Sodium Hydroxide, g/L as Na ₂ O	Sodium Carbonate, g/L as Na ₂ O	Total Titratable Alkali, g/L as Na ₂ O
Venturi sump	9.75	110	5.10	0.01	6.8	52.9	59.7
Teller TRS sump	9.30	29.4	6.40	0.18	10.4	19.8	30.4
Heat recovery sump	7.86	0.42	0.04	0.06	0.20	0.00	0.26

III. Analysis of sump compositions in the Teller scrubber. Results were averaged from triplicate determinations.

Effectiveness of TRS removal by the scrubber

For effective TRS scrubbing, the pH in the venturi and TRS scrubber sumps should be maintained at or above pH 9. Samples from the venturi sump, Teller TRS sump, and heat recovery sump were obtained and analyzed during July–September 2014. Results of the analyses are presented in **Table III**. Analyses were performed by ion chromatography according to TAPPI T624 cm-00 “Analysis of soda and sulfate white and green liquors” and by the ABC test method [10]. The results show that the pH and total titratable alkali (TTA) content are highest in the venturi sump where the flue gases enter first and slightly lower in the Teller TRS scrubber. The pH in both sumps was well in excess of 9.0. The recirculated scrubbing liquor from both sumps would perform acceptably for H₂S removal.

The TRS gases entering the Teller scrubber system are carried with the recovery boiler flue gas as it exits the cyclone evaporators. The concentrations of each of the four TRS components are determined by the amounts of each that are re-

Location	Hydrogen Sulfide, ppm*	Methyl Mercaptan, ppm**	Dimethyl Sulfide, ppm**	Dimethyl Disulfide, ppm***	Total, ppm
Teller inlet	95	7.0	0.0	1.3	103.3

* = Measured with both Draeger tubes and with gas chromatography from samples collected in Tedlar bags; ** = measured with Draeger tubes on July 7 and 11, 2014; *** = measured with gas chromatography from samples collected in Tedlar bags on September 2, 2014.

IV. Gas concentrations of the four TRS compounds measured at Teller scrubber inlet.

Data Source	This Study*	1973 Study**
Component	Vol%	Vol%
Hydrogen sulfide (H ₂ S)	92.1	78.9
Methyl mercaptan (CH ₃ SH; MM)	6.8	10.5
Dimethyl sulfide (CH ₃ SCH ₃ ; DMS)	0.0	5.3
Dimethyl disulfide (CH ₃ S ₂ CH ₃ ; DMDS)	1.2	5.3

* = Measured at the Teller system gas inlet; ** = measured at the direct contact evaporator outlet.

V. Comparison of the distribution of TRS compounds measured at equivalent locations at two kraft pulp mills.

leased from the black liquor by contact with the hot flue gas. Some of the Na₂S and NaHS in black liquor are converted to H₂S in the cyclones by reaction with CO₂, an acid gas in the flue gas. Dimethyl sulfide and DMDS, both very volatile species at the liquor temperature in the cyclones, evaporate from black liquor. Methyl mercaptan also evaporates and can be oxidized to the more volatile DMDS.

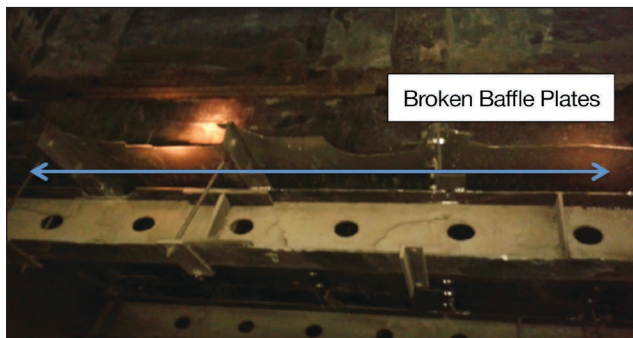
Measurements of the concentrations of all four TRS compounds at the Teller system inlet (**Table IV**) were made July–September 2014 during the period when the mill's TRS emissions were out of compliance. The intent of these measurements was (a) to determine whether poor performance of the Teller scrubber was the reason for TRS emissions being out of compliance and (b) if so, which of the TRS compounds were not being removed effectively by the Teller scrubber.

Table V shows a comparison of the distribution of TRS compounds measured at equivalent locations at two kraft pulp mills: the Teller scrubber system inlet in this study and at the direct contact evaporator outlet at a Swedish mill [11].

On occasions when TRS emissions exceeded 15 ppm at the recovery stack, sodium hypochlorite was applied at the Teller stack just before the flue gases exit to the atmosphere as needed to limit TRS emissions. Sodium hypochlorite is an effective oxidant for DMS and DMDS [9] and can also oxidize H₂S. However it is corrosive. It damages the stack and adds additional cost to the operation. It is used as a TRS control method of last resort by the mill.



6. Broken tellerets in the TRS sump packed bed.

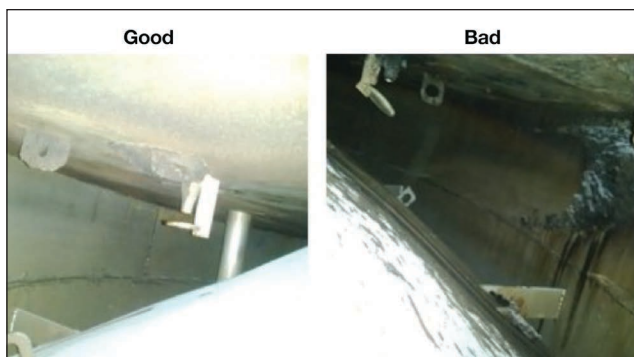


7. Broken baffles in the Teller scrubber.

High TRS emissions due to equipment problems

Several equipment-related problems with the Teller scrubbers and cyclone evaporators were observed during the June 2014 shutdown. All of these problems would have contributed to excessive TRS emissions by reducing the efficiency of the TRS scrubber in removing H₂S. The following are the identified equipment-related problems:

- **Plugged spray nozzles.** The majority of the scrubbing solution spray nozzles in the Teller TRS section were plugged.
- **Damaged TRS scrubber packing.** The bed height of the tellerets varied across the scrubber with a low bed height observed in 40% of the openings. Tellerets were found to be broken, as shown in **Fig. 6**. The damage may be due to temperature shock in some of the beds.



8. Bent splash liquor plates.

- *Plugged venturi section headers.*
- *Broken baffle plates.* During the inspection in the October mill shutdown, it was observed that the Teller scrubber TRS sump had broken baffle plates on the east side across the whole width of the scrubber (**Fig. 7**). The broken baffles result in short-circuiting of TRS gases, leading to inefficient scrubbing. This can put the mill out of environmental compliance.
- *Air leaks.* The cyclone evaporators had a few leaks due to corrosion that led to tramp air being entrained in the system. Dimethyl disulfide is generated by oxidation of MM, so contact of oxygen with the cyclone liquor formed DMDS. Because DMDS is not scrubbed in the Teller scrubber, hypochlorite had to be used to oxidize DMDS and keep the mill within environmental compliance.
- *Bent liquor splash plates in cyclones.* Bent splash plates cause dry spots in cyclone evaporators and nonuniform wetting of the umbrella. This leads to drying and pyrolysis of black liquor, and subsequent emission of organosulfur gases (**Fig. 8**).
- *Condition of the cyclones.* When the cyclones are fouled, contact between CO_2 in the flue gas and the black liquor is not uniform. This can cause low pH of black liquor locally within the cyclones, increasing emissions of H_2S and organosulfur gases.

RECOMMENDATIONS

Recovery boiler and Teller scrubber

The following actions are recommended for the recovery boiler and Teller scrubber:

- Run excess air to the recovery boiler between 2%–3.5% to maintain $\text{CO} < 200$ ppm.
- Monitor the electrostatic precipitator (ESP) ash pH closely. Optimal pH range is between 10 and 11. Having pH values in this range means that the bed temperature is hot enough and these pH values result from Na_2CO_3 being generated in the recovery boiler [1]. The mill's ESP ash values of pH 10.9 and pH 11.1 were measured in July and August 2014.
- Inspect the packed bed height in the Teller scrubber during area shutdowns and repack with tellerets to an appropriate level in case of lower bed height.

- Make sure that the NCG line from multiple-effect evaporators to the Teller scrubber system is completely shut because these NCGs contain mostly organosulfur gases.
- Include analyses of Teller scrubber gases for H_2S , MM, DMS, and DMDS in the biweekly laboratory tests. These test results will help in determining the performance of the Teller scrubber and hence in indicating whether a TRS issue is related to the scrubber performance or any other process or mechanical issues upstream of the scrubber.

Cyclone evaporator

Organosulfur gases can be produced in direct contact evaporators if temperatures are high enough to dry and pyrolyze some of the black liquor solids. If cyclones are fouled, the contact between CO_2 and flue gases is not uniform (high residence time), resulting in lower pH of black liquor and subsequent emission of H_2S and MM.

A high evaporation load in the cyclones and the number of cyclone inlet sprays also seem to cause higher TRS generation. This can be explained by the fact that lower multiple-effect evaporator product liquor solids lead to higher black liquor droplet surface area in cyclone evaporators and hence high TRS. The following actions are recommended for the cyclone evaporator:

- Run higher multiple-effect evaporator product solids concentration but stay at 2% dry black liquor solids below the first crystallization point for carbonate-sulfate salts to avoid scaling.
- Run with a lower number of inlet sprays if TRS emissions are high.
- Run the cyclone inlet at $\text{pH} > 12.1$. If required, add caustic solution to the black liquor going to the cyclone evaporators.
- Inspect the liquor splash plates frequently for damage, and repair as needed.
- Monitor cyclone fouling closely.
- Monitor cyclone leaks to reduce entrainment of tramp air.

CONCLUSIONS

This study addressed challenges faced at a Canadian dissolving grade pulp mill in 2014 with meeting its TRS gases emission limit of 15 ppm, as governed by the regulations under New Brunswick's Clean Air Act. The TRS compounds emitted by the mill are H_2S , MM, DMS, and DMDS. The amounts of each entering the Teller TRS scrubber system were no higher during the period of excessive emissions from June–October 2014, and the TRS scrubber should have been capable of meeting the mill's TRS emissions standard if the scrubber were in good repair and operating as designed.

An inspection of the Teller process equipment found that TRS emissions from it were caused by two groups of factors. One included process operating problems that included

plugged spray nozzles that deliver scrubbing liquor to the TRS absorber bed, damaged TRS scrubber packing, and plugged venturi section headers.

The second group of factors included mechanical equipment failures. These included broken baffle plates in the TRS scrubber sump, air leaks in the cyclone evaporators, bent liquor splash plates in the cyclone evaporators, and fouling of the cyclone evaporators with dried black liquor. These mechanical issues were fixed during the October 2014 mill annual outage. After startup, the TRS issues experienced in the previous 4 months vanished. **TJ**

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ABOUT THE AUTHORS

Capacity limitations resulting from TRS emissions and increased chemical cost to manage TRS emissions at the concerned mill led to research of this topic. This work is based on a mill study that was carried out to troubleshoot TRS issues. It is a unique study of a kraft dissolving pulp mill that uses direct contact evaporators and a teller scrubber.

The most difficult aspect of the research was to narrow down the list of multiple variables that effected TRS emissions. We addressed this issue by a thorough literature survey and chemical analysis of gases. Surprisingly, we discovered that although the problem seems complicated and a strong focus on pulping chemistry and scrubbing chemistry was needed, sometimes a less complicated mechanical issue could be the source of the problem.

Mills can benefit from the extensive literature on TRS generation, collection, and destruction and the various troubleshooting steps involved in identifying the problem. Our next step is to generate a standard



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operating procedure for the mill so that the root cause of the problem can be identified quickly if the problem arises again.

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