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DISSOLVED GAS MEASUREMENTS FOR BORES IN THE SOUTH EASTERN PART OF THE SURAT BASIN (GREAT ARTESIAN BASIN, NORTH EASTERN NSW)

A report to the NSW Department of Mineral Resources

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1. SUMMARY

Gases from 23 water bores sampled from the Goondiwindi district (NSW) were studied to determine their origin. The objective of this type of survey was to use the bores as a hydrocarbon exploration tool. These bores lie in the southeastern most part of the Surat Basin, which is part of the Great Artesian Basin (GAB) aquifer. Gas compositions were determined for all 23 headspace samples. In 21 of these samples, methane (CH_4) was the only hydrocarbon gas present, while the remaining two contained both CH_4 and traces of ethane (C_2H_6). Other gaseous components dissolved in these waters were carbon dioxide (CO_2) and nitrogen. Stable carbon isotope analyses were conducted on the CH_4, C_2H_6 and CO_2 components from several bores that were selected on the basis of gas concentration and their positioning with respect to the Goondiwindi Fault. These analyses indicated that all the samples contained dry gas (wet gas index > 0.996) with isotopically depleted CH_4 signatures ($\delta^{13}C$ averaging –58.3‰) consistent with biogenic input. Four of the samples were taken on the eastern side of the Goondiwindi Fault in order to assess the possibility of gases from different sources. Two of these samples indicated possible CH_4 contributions of a thermogenic origin with $\delta^{13}C$ values of –54.7 and –55.3‰. Some bores sampled in earlier studies (1995 to 1999) were re-visited and comparison with current data indicated general consistencies. Some differences amongst duplicate analyses highlighted the need for some changes in the way these type of samples are handled.

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1. INTRODUCTION

Water samples (23) obtained from artesian bores were collected by the New South Wales Department of Mineral Resources (NSW DMR) for gas compositional and carbon isotopic analysis. The aim of the sampling survey was to add to the existing database of gases dissolved in water bores already held by the NSW DMR and to identify the locations of thermogenic gas (i.e. hydrocarbon) anomalies. Anomalies are possible indicators of leakage from hydrocarbon reservoirs.

The combined analytical techniques (i.e. wet gas ratios and stable carbon isotopes) were used to characterise the gases. Methane (CH_4) samples with bacterial origins have diagnostically depleted stable carbon isotope ($\delta^{13}C$) values in the broad range –50 to –120‰ (Whiticar, 1994). Usually these types of gases are also compositionally dry with wet gas indices or $C_1/(C_1\text{-}nC_5)$ far exceeding 0.98 (Bernard, 1978; Hunt, 1996). Furthermore these techniques can be used as correlation tools. Bores on either side of the Goondiwindi Fault, for example, could be sourced from different coals (D. Alder, pers. comm.), and these differences may become evident in the gas compositions.

2. ANALYTICAL METHODS

2.1 Sample collection

Water samples were collected directly from bore flumes into 1000 mL flasks and immediately sealed with septa (silicone-rubber) fitted caps. During sample collection and transfer to the CSIRO laboratories (a period of more than 10 days), gas equilibrated between the headspace and water enabling the sub-sampling of aliquots from the (approximately) 135 mL of headspace. Samples were received in a chilled state and were immediately placed in cold storage ($3^{o}C$) prior to analysis. Samples were allowed to reach room temperature prior to analysis (near STP conditions).

2.2 Gas compositions

Natural gas compositions were determined on a SRI 8610 gas chromatograph (GC) fitted with a thermal conductivity detector (TCD) and a flame ionisation detector (FID) connected in series. Gas samples were introduced via a 1 mL sample loop. The C_{2+} hydrocarbons (ethane and higher) and carbon dioxide (CO_2) were resolved on a 1.5 m x 3 mm (od) stainless steel column packed with silica gel. The oxygen peak (which includes argon), nitrogen (N_2) and CH_4 were resolved on a 1 m x 3 mm (od) stainless steel column packed with 5Å molecular sieve medium. The temperature of the molecular sieve column was held constant at 60°C. The silica gel column was programmed from 40°C (5 minutes) to 140°C at 10°C / min, held for 6 minutes then heated to 220°C at 10°C / min and finally held for 11 minutes. The non-hydrocarbon gases and CH_4 (>1%) were detected by TCD. Abundances were calculated from peak areas using weight factors based on the relative thermal conductivities of the individual gases which were cross checked with a BOC standard. The FID detector was used for quantifying hydrocarbon concentrations below 1%.

2.3 Stable carbon isotope ($\delta^{13}C$) analysis of gases

Stable carbon isotope analysis of the CH_4 and CO_2 was achieved by (i) preparative chromatographic separation, conversion, and trapping of individual gas species on a custom-built system using a Poropak column (4 m x 3 mm od) and a cupric oxide furnace heated to 900°C, (ii) sample purification and drying by cryo-distillation at -78°C and, (iii) isotope ratio mass spectrometry (IRMS) for the individual compounds by dual inlet measurement on a Finnigan MAT 252. The carbon analytical system is routinely checked against the international natural gas materials NGS #1 and NGS #2, and laboratory standards. The stable carbon isotope compositions are expressed in parts per thousand (‰) relative to Vienna PeeDee Belemnite (VPDB), according to the expression:

$$\delta^{13}\text{C ‰} = 1000 \times \frac{({}^{13}\text{C}/{}^{12}\text{C}_{\text{ sample}} - {}^{13}\text{C}/{}^{12}\text{C}_{\text{ reference}})}{{}^{13}\text{C}/{}^{12}\text{C}_{\text{ reference}}}$$

Isotopic precision in the gases for the methane and ethane standard is 0.2‰ and for CO_2 0.3‰.

2.4 Effect of time and temperature on carbon isotope values

During the preparation and analysis for the carbon isotopic compositions of the CH_4 and CO_2 components from these bore waters, preliminary results suggested that these samples were being affected by some process, possibly biodegradation. In the first four samples analysed, duplicate isotope analyses were carried out within 24 hours of the first analyses (Table 1) with samples being allowed to remain at room temperature over this period. The remaining samples were then analysed isotopically in duplicate within two hours of each other. Three of these initial four samples and an additional sample were subsequently removed from refrigeration, held at room temperature for 72 hours and re-analysed in order to discount biodegradation of gases at room temperature. The first set of isotopic analyses have been used during the interpretation and discussion of the bores.

Table 1: Time interval between samples tested isotopically.

	Timing of duplicate isotope analyses in hours		
Telleraga-2		2	
Neargo-1		2	72
Boobora-2		2	
Boolooroo		2	
Milroy		2	
Saleyards	24		72
Kiga-1		2	
Marlow		2	
Allendale-1	24		72
Boonal	24		72
Allendale-2	24		
Osterley		2	

3. RESULTS AND DISCUSSION

3.1 Gas composition

The chemical compositions of the 23 headspace gases sampled are listed in Table 2. Due to the sampling method, air is always present as a contaminant. Abundances of non-atmospheric components range considerably, depending on the amounts of dissolved gases present. Thus, where the amounts of dissolved gases are low, the level of the air component is much more apparent.

3.1.1 Nitrogen and carbon dioxide

Good chromatography ensured that N_2 was separated from the other main air component (combined O_2 + Ar), allowing a reliable determination to be made of the amounts of N_2 in the sample headspaces (see as received analyses in Table 2). The sample from Saleyards has the least amount of air present and provides the best guide in assessing the amount of N_2 derived from the aquifer. Atmospheric air has the basic composition: 78.08% N_2, 20.95% O_2 and 0.93% Ar (Tiratsoo, 1972). Based on this it is then estimated that around half of the gas in Saleyards is composed of N_2 which has originated from a geological (perhaps thermogenic) source. N_2 is probably the most abundant gas in most of these samples, followed in many cases by CH_4 and CO_2.

CO_2 abundance varies moderately from 0.1 to 3.1% of the headspace gas. At low levels (e.g. <0.1%) the amount of atmospheric CO_2 contamination (>0.035%) may become an important consideration. Its compositional variation is not as great as CH_4 which is probably because of its higher solubility in water. Another possibility is that the source of this CO_2 could have a greater spatial uniformity. These sources could be inorganic CO_2 from either the mantle or carbonate decomposition, or organic CO_2 from either dissolved organic compounds in the artesian system or the maturation of source rocks and coals

3.1.2 Hydrocarbon gases

The gas compositions are tabulated in Table 2 along with their wet gas indices, defined here as $C_1/(C_1\text{-}nC_5)$. The CH_4 headspace concentrations averaged 5% with the highest at 17.7%. Most (14) of the samples were below 0.1% or 1000 parts per million by volume (PPMV) and these lay to the west of the Goondiwindi Fault. The sampled bores located to the east of the Goondiwindi Fault all have relatively high levels of CH_4 (8.26 to 17.5%). Clearly almost all the studied samples have a wet gas index of 1.0 since only CH_4 was detected. The exceptions are Boolooroo and Kiga-1 which have indices of 0.996 and 0.9999 respectively due to the trace amounts of ethane present. All the values are regarded as extremely dry. In general such indices can be associated with either biogenic CH_4 production (Whiticar, 1994), extremely high source rock maturities (James, 1983) or some coals (Smith and Pallasser, 1996). In order to narrow the possible origin it was necessary to combine these data with isotopic information (see Section 3.2.1).

Table 2: As received headspace gas compositions of the water bores studied.

Bore No. **Name**	4405 Naroa	4378 Mungyen	4378 Mungyen Sample 2	4495 Telleraga-2	4385 Neargo-1	Moomin	Boobora-2	Bunaba-2	4345 Kiga-3	4413 New Yarrawa	4578 Tulloona-2	4023 Boolooroo
Sampling Date	27.4.01	26.4.01	26.4.01	27.4.01	-	27.4.01	23.4.01	26.4.01	22.4.01	22.4.01	23.4.01	24.4.01
Analysis Date	11.5.01	11.5.01	11.5.01	11.5.01	11.5.01	11.5.01	11.5.01	15.5.01	15.5.01	15.5.01	15.5.01	16.5.01
CH_4 PPMV	3455	7533	4353	7162	7893	5018	32900	3493	6572	2911	2243	48900
C_2H_6 PPMV	n.d.	n.d.	n.d.	sl. tr.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	191
CO_2 PPMV	3645	2832	2925	2904	4232	2688	1899	1897	1018	2013	1273	1518
O_2+Ar Mol %	18.7	17.6	18.9	17.1	17.9	19.0	19.0	20.7	16.7	18.0	18.2	18.2
N_2 Mol %	80.6	81.3	80.4	81.9	80.9	80.2	77.6	78.7	82.6	81.5	81.5	76.7
$(C_1/C_1\text{-}nC_5)$	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	0.996

Bore No. **Name**	4340 Milroy	39380 Toomelah	10778 Saleyards	4288 Kiga-1	4342 Marlow	24784 Allendale-1	10063 Boggabilla	15926 Galvin Park	6574 Boonal	22902 Allendale-2	1326 Osterley
Sampling Date	23.4.01		19.4.01	21.4.01	20.4.01	19.4.01		20.4.01		20.4.01	22.4.01
Analysis Date	16.5.01	16.5.01	16.5.01	16.5.01	16.5.01	17.5.01	17.5.01	17.5.01	17.5.01	17.5.01	17.5.01
CH_4 PPMV	592	4289	175000	176700	104000	107400	1477	6131	83600	82600	140600
C_2H_6 PPMV	n.d.	n.d.	n.d.	0.0025	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
CO_2 PPMV	1077	1192	30500	14800	15500	4500	1231	1605	2706	2567	2681
O_2+Ar Mol %	17.4	17.4	8.1	14.1	16.8	12.7	16.6	16.3	15.5	14.3	16.1
N_2 Mol %	82.4	82.1	71.4	66.7	71.3	76.1	83.1	82.9	75.8	77.2	69.6
$(C_1/C_1\text{-}nC_5)$	1.00	1.00	1.00	0.999	1.00	1.00	1.00	1.00	1.00	1.00	1.00

n.d. : not detected
sl. tr. : <5 PPMV

3.2 Stable carbon isotope analyses

3.2.1 Methane

The isotopic compositions of CH_4 ranged between –68.0 and –30.3‰ VPDB (first analysis quoted, Table 3). The isotopically depleted values are consistent with gases sampled from other bores reported for previous NSW DMR surveys (Pallasser et al., 1995; Pallasser, 1996, 1997, 1998, 2000) which were mostly regarded as having a biogenic origin (Figure 1).

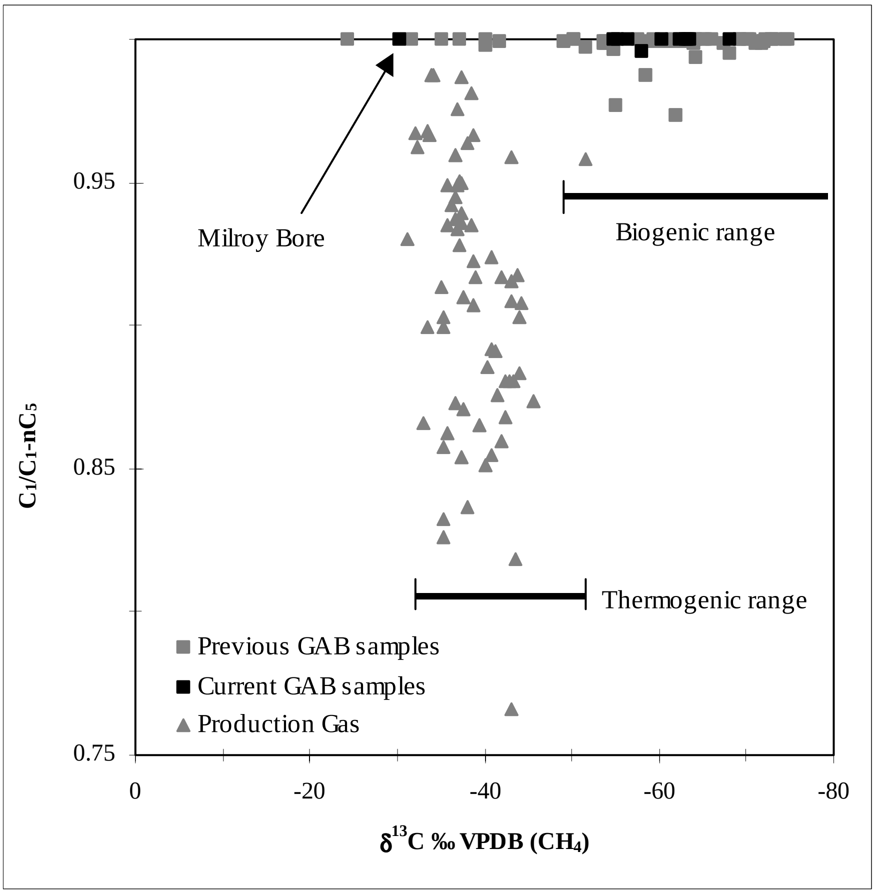

Figure 1: Modified Bernard Plot where the wet gas index or $C_1/(C_1$-$nC_5)$ is compared to the stable carbon isotope composition of the related CH_4. The published ranges for thermogenic and biogenic CH_4 are also marked (Whiticar, 1994).

The water bore gases clearly fall in a separate field to production gases from around Australia (predominantly thermogenic in origin). The thermogenic gases mostly have a narrow isotopic range which is comparatively enriched by some 20‰ to most bore water gases, while generally being compositionally much wetter as well. Where the $\delta^{13}C$ value of CH_4 from some previously studied water bores fall within the thermogenic range, this might indicate

Table 3: Summary of stable carbon isotopic analyses including duplicate analyses.

Bore No.	4495	4385		4023	4340	10778	4288	4342	24784	6574	22902	1326
Name	Telleraga-2	Neargo-1	Boobora-2	Boolooroo	Milroy	Saleyards	Kiga-1	Marlow	Allendale-1	Boonal	Allendale-2	Osterley
$\delta^{13}C$ ‰ VPDB												
CH_4*	–64.2	–63.1	–56.5	–58.1	–30.3	–54.7	–62.3	–63.5	–60.3	–68.0	–55.3	–63.3
CH_4	–62.4	–62.7	–55.8	–58.1	–31.5	–55.1	–62.4	–63.3	–63.1	–69.2	–57.6	–63.3
CH_4 (72 hours)		–62.1				–49.7			–59.9	–67.5		
CO_2*	–15.9	–14.8	–9.1	–16.6	–18.1	–8.1	–10.2	–12.5	–13.0	–10.3		–8.2
CO_2	–15.3	–14.3	–8.8	–16.0	–18.5	–7.6	–10.5	–12.3	–9.2	–8.6		–7.3
CO_2 (72 hours)		–14.1				–8.1			–9.4	–9.9		
C_2H_6*				–36.6								
C_2H_6				–36.3								

*****The first isotope analyses are used for interpretation and discussion.

possible leakage of hydrocarbons from a deeper thermogenic source. In the current study, Milroy (Bore No. 4340) has the most enriched $\delta^{13}C$ value at –30.3‰ which is 28‰ heavier than the average CH_4 isotopic composition found in the current analyses. Such a composition would usually be regarded as very mature or possibly coal related. However, this value also stands out because it has by far the lowest CH_4 concentration of all the currently analysed samples (0.0592%). In addition the samples with similarly enriched CH_4 values from previous surveys that are plotted on Figure 1 also have much greater CH_4 concentrations, between 2.1 and 11.4%. This raises uncertainty about the integrity of the Milroy data.

Boolooroo Bore (closest to Moree) contains 191 PPMV of C_2H_6, an encouraging sign for thermogenic hydrocarbon prospectivity. However, the associated isotopic composition (–58.1‰) of the abundant (4.8%) CH_4 appears more biogenic (Figure 1). The $\delta^{13}C$ values for Boobora-2 (–56.5‰), Saleyards (–54.7‰) and Allendale-2 (–55.3‰) are tending towards compositions intermediate between biogenic and thermogenic. These values could indicate contributions from thermogenic sources, especially Saleyards with its high abundance of 17.5‰ of CH_4.

3.2.2 Carbon dioxide

The isotopic compositions of CO_2 shown in Table 3 range from –18.8 to –8.1‰ which covers a range of origins. The concentrations of CO_2 have been plotted against their $\delta^{13}C$ value on Figure 2. Atmospheric CO_2 has an isotopic composition of –7‰ VPDB and where CO_2 from the bore water is low, air contamination may have an influence on the final isotopic composition. A number of samples with low (< 0.3%) to moderate (3.1%) CO_2 concentrations have isotopic signatures in a range consistent with an inorganic (mantle degassing or carbonate decomposition) source of the CO_2, although an organic origin cannot be ruled out due to secondary biogenic processes. The CO_2 from Milroy had an isotopic composition (–18.1‰) most reflective of organic origin.

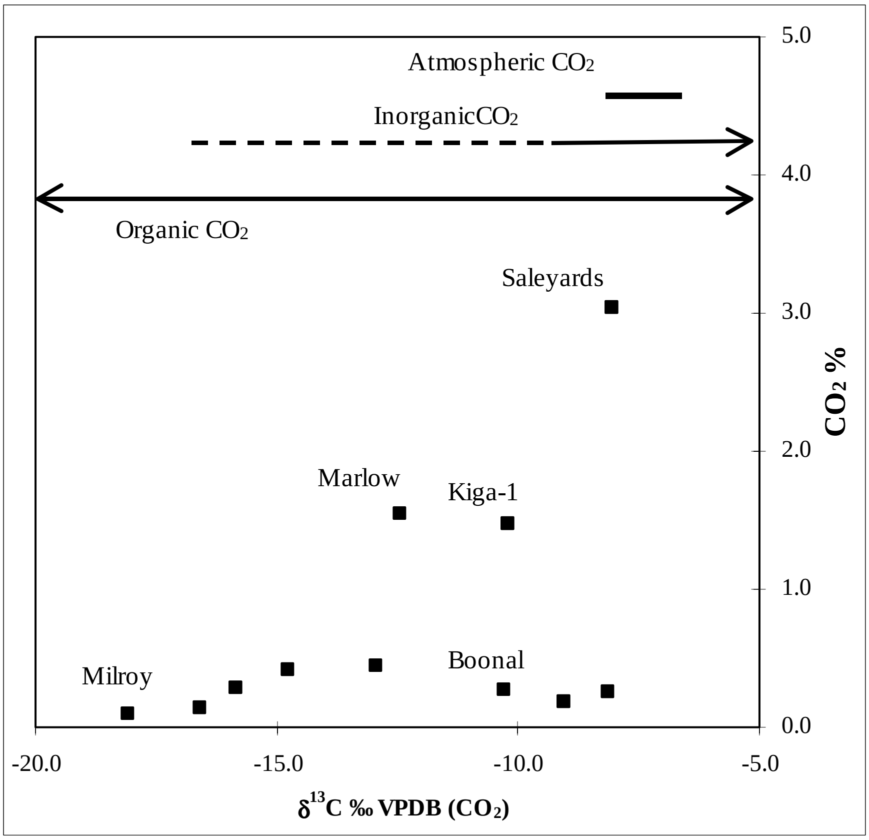

Figure 2: Plot of δ^{13}C and the concentration of CO_2.

3.3 Effect of time and temperature on carbon isotope values

Duplicate isotopic analyses were initially carried out within a time interval of 24 hours while samples were held at room temperature (Table 1).The resulting isotopic compositions varied by up to nearly 4‰ with the CH_4 showing isotopic depletion (1-3‰) and the CO_2 isotopic enrichment (0.5-3.8‰) in three of the four consecutively performed analyses as shown on Table 3. These results raised concerns about possible alteration effects, physical or biogenic, occurring in the sample bottles, and that these could be promoted by warmer conditions (i.e room temperature). Therefore subsequent duplicate analyses on the remaining samples were carried out within 2 hours of one another after removal from refrigeration (Tables 1 and 3). These duplicate analyses vary by up to 1.8‰ for the CH_4 and 0.9‰ for the CO_2.

To assess the likelihood of biological alteration, three of the four samples held at room temperature for 24 hours and an additional sample (Neargo-1) were analysed again after these samples were maintained at room temperature for 72 hours. However, instead of showing a continuation of the earlier trends found for CH_4 and CO_2, the new values generally coincided with either the first or second analysis, with the exception of Saleyards, which became 5‰ enriched. From the variable isotopic trends, it is difficult to ascribe the changes to leakage from the bottles occurring between sub-sampling. Based on all the isotope data it is unclear whether the cause of the isotopic variation for any sample is physical, biological or just due to greater errors associated with this type of sample and analysis.

3.4 Comparison of current analyses with previous work

During the current survey, water samples were taken from bores in the areas adjacent to previously studied bores (D. Alder pers. comm.) (Table 4). Bryanungra was studied previously (in 1999), and lies in the same general area as Boobora-2, Milroy, Marlow and Osterley. While there are some gas compositional differences, the $\delta^{13}C$ values for CH_4 from Marlow and Osterley are fairly close to Bryanungra (within <0.6‰). Conversely, the CH_4 from Milroy with the value –30.3‰ was isotopically completely different. However, the CH_4 value from Milroy is held in some doubt (see Section 3.2.1) and should be used with caution in any comparison. Further isotopic characterisation was sought for the Krui area so the nearby bores, Telleraga-2 and Neargo-1 were studied in this round of analyses. The abundance of CH_4 was much greater in Telleraga-2 and Neargo-1 than Krui, while $\delta^{13}C$ values for the CO_2 varied by 6‰. The bores which were re-sampled (New Yarrawa, Tulloona-2, Milroy, Marlow and Kiga) had compositions in the same general range as found previously with the exception of Marlow where the CH_4 was nearly four times that determined in 1999. The CH_4 from the currently analysed Kiga-1 was also greater (an increase of 70,000 PPMV) as well as being 4‰ depleted in $\delta^{13}C$ compared to an analysis in 1998. This is an indication that some gas could have been lost in the earlier sampling survey along with isotopic fractionation. The CH_4 content in other bores varied somewhat but is not regarded as significant where the concentration range is below 10,000 PPMV.

Table 4: Gas compositional, and where available, isotopic data from previous and current surveys (values expressed in PPMV).

Sampling Year	**Name**	**Bore No.**	**CH_4 PPM**	**C_2H_6 PPM**	**C_3H_8 PPM**	**CO_2 PPM**	**CH_4 $\delta^{13}C$ ‰ V PDB**	**CO_2 $\delta^{13}C$ ‰ V PDB**
1999	Bryanungra	4034	506900			3300	-63.9	-8.8
2001	Boobora-2		32900			1899	-56.5	-9.1
1999	Milroy	4340	452			1500		
2001	Milroy	4340	592			1077	-30.3	-18.1
1999	Marlow	4342	26800	pos. tr		16700		
2001	Marlow	4342	104000			15500	-63.5	-12.5
2001	Osterley	1326	140600			2681	-63.3	-8.2
1995	Krui		43	0.1	tr	3345		-21.3
1996	Telleraga-2	4495	5200	7.2	0.9	3400		
2001	Telleraga-2	4495	7162	sl. tr		2904	-64.2	-15.9
2001	Neargo-1	4385	7893			4232	-63.1	-14.8
1996	New Yarrawa	4413	5825	3		3100		
2001	New Yarrawa	4413	2911			2013		
1999	Tulloona-2	4578	2184			900		
2001	Tulloona-2	4578	2243			1273		
1996	Kiga-1	4288	95700	26		20800		
1998	Kiga-1	4288	97100	12.8	0.03	13000	-58.0	
2001	Kiga-1	4288	176700	25		14800	-62.3	-10.2
1999	Kiga-2	4332	18800			3100		
1996	Kiga-3	4345	3400	0.7		3000		
2001	**Kiga-3**	4345	6572			1018		

3.5 Gas characteristics east of the Goondiwindi fault

The bores Saleyards, Allendale-1 and -2 and Boonal are located east of the Goondiwindi Fault and could be influenced by other coal sources (D. Alder pers. comm.). The sequence deepens in this eastern section, although an additional younger coal appears at a more shallow depth, not present in the western section. Any differences in either gas or isotopic composition would indicate possible separate sources and/or lack of gas migration across the fault. The bores closest to the fault on the western side, Marlow and Kiga, have been tabulated along with the four bores on the eastern side (Table 5)., The eastern bores are fairly comparable in CH_4 abundance, while the CO_2 concentrations are lower with the exception of Saleyards. There is some overlap in the carbon isotopic compositions of the CO_2 and also the CH_4. The most apparent difference is that the two bores, Saleyards and Allendale-2 (eastern side of fault) are isotopically heavier by more than 5‰ and could be interpreted as contribution from another or additional source.

Table 5: Bores located closest to the east and west of the Goondiwindi Fault.

Name	**Bore No.**	**CH_4 PPM**	**C_2H_6 PPM**	**CO_2 PPM**	**CH_4 $\delta^{13}C$ ‰ V PDB**	**CO_2 $\delta^{13}C$ ‰ V PDB**
Bores closest to fault on eastern side						
Saleyards	10778	175000		30500	-54.7	-8.1
Allendale-1	24784	107400		4500	-60.3	-13.0
Boonal	6574	83600		2706	-68.0	-10.3
Allendale-2	22902	82600		2567	-55.3	
Bores closest to fault on western side						
Marlow	4342	104000		15500	-63.5	-12.5
Kiga-1	4288	176700	25	14800	-62.3	-10.2

4. CONCLUSIONS AND RECOMMENDATIONS

1. The gases from the bores studied in the current survey primarily comprise CH_4 with a biogenic carbon isotope signature. While the amounts of gas hydrocarbons are at times quite significant, the gases are predominantly composed of CH_4 with occasional traces of higher hydrocarbons. Such dry gas compositions support a biological generation mechanism as indicated by the isotopic study. However this does not preclude leaking thermogenic hydrocarbons from oil pools or coals being the original source of these gases.

2. Bores that are interpreted to contain some thermogenic CH_4 are Boobora-2, Saleyards and Allendale-2. The CH_4 rich gas from Boolooroo contains traces of C_2H_6 and could fall into this same category. Milroy shows some isotopic evidence of being strongly thermogenic, however abundances of material are very low.

3. The bores which lie on the eastern section of the Goondiwindi Fault have lower dissolved CO_2, except for Saleyards. Compositions from Saleyards and Allendale-2 are more likely to represent direct CH_4 contribution from thermogenic sources, due to their intermediate $\delta^{13}C$ values, high concentrations and the greater potential for up-dip migration from further north in the Surat Basin.

4. Data collected during the current round of analyses (2001) are fairly consistent with the previously gathered compositional and isotopic data, but there are some exceptions. These may be related to analytical variations as highlighted during the current isotopic work and/or loss of gases during/after sample collection.

5. Some possible analytical issues were raised by the work which suggest that the use of anti-bacterial agents in any later sampling should be employed to avoid possible alteration of the gas samples. Therefore it is recommended that future sampling of bore waters should involve the pre-addition of an effective bactericide such as mercuric chloride so that potential bacterial alteration is eliminated altogether. In addition, study of multiple samples from the same bore would be useful in determining the reproducibility of sampling, storage and analysis, the results of which would have implications for the relative errors in this work.

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