

高含硫气藏水合物形成机理的实验研究

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摘 要 近年来随着川东北罗家寨、铁山坡等高含硫气藏的相继勘探成功, 这些气藏将成为川渝地区重要的接替区块。对高含硫气藏水合物机理进行了实验研究, 并对实验原理、实验方法、实验现象等进行了详细论述。首次得出了高含硫气藏水合物在实验条件下是按照天然气的比例以混合物整体形式同时与水分子结合形成的, 而不是按优先选择原则逐个顺序形成的结论。

关键词 含硫气体 天然气 水合物 硫化氢 形成机理 生成条件

近年来, 随着高含硫气藏的不断勘探和开发, 特别是目前川东北地区渡口河、罗家寨、铁山坡气田飞仙关组气藏的相继勘探成功, 对此类气藏的气态特征以及水合物形成的研究日益受到广大科研工作者

的关注和重视。由于国内在这方面的研究没有较为完善的理论, 所以, 开展对罗家寨、铁山坡等高含硫气藏的水合物形成的研究, 一方面可以更深入地完善和丰富此领域的理论, 另一方面也可作为开发方案

脱水和制 O_2 装置即可, 而不必再配套建设硫磺回收、尾气处理装置, 节资节能效果显而易见。同样地, 在处理量为 $600 \times 10^4 m^3/d$ 不变的情况下, 如果气田原料气 H_2S 浓度提高到 $12\% \sim 15\%$, 硫回收与尾气装置采用 $30\% \sim 45\%$ 富氧空气就可满足要求, 大大提高了装置的操作弹性和经济性。

几点建议

笔者提出二级 Claus、串级 Scot 及富氧 Claus 等设想, 旨在通过优选方案, 力求工艺简炼, 技术先进成熟, 运行安全可靠, 降低工程投资和操作成本, 提高工程建设项目整体经济效益。

(1) 对于高含硫天然气净化厂, 从安全生产角度而言, 装置和设备数量宜少而精。如果委托外方做基础设计, 设计优化后成套引进关键主体装置和设备, 则既可保证总体投资不至太高, 又能提高装置性能和安全可靠性, 减少非计划停车及检修时间^[2], 最终确保工厂有较高的开工率。

(2) 鉴于工艺路线的复杂性, 实施建设时宜在控制好节奏的前提下按两步法对外招标: 即先进行工艺技术方案招标, 博采众长, 确保方案质量; 待评标优选出方案后, 再进行商务招标, 选择业界内实力雄

厚、富有经验、业绩良好、报价合理的工程公司提供工程设计和成套供货服务。

(3) 根据川东北气区的开发建设总体规划, 宜先引进成套硫磺回收、尾气处理装置, 确保首套高含硫天然气净化装置安全、可靠、平稳运行, 以保证东输工程的如期顺利实施, 同时也为将来实现国产化积累经验。

(4) 鉴于其他净化厂的经验, 在下一步工作中尚须仔细考虑有关工厂污水处理回用与达标排放、硫磺成型方式与输运、工厂高低压蒸汽平衡控制、原料气与胺液的过滤分离等技术经济问题。

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的制定和地面工程建设提供理论依据和数据保障,为搞好该区块开发规划,分析评价该区块资源潜力,提高新区整体勘探开发效益,以及对“十五”西南油气田分公司天然气产量稳步发展都具有十分重要的意义。

到目前为止,尚未见到按硫化氢含量对硫化氢油气藏进行完整的分类。但是,分析国外资料可以看出:通常把硫化氢含量为 2%~5%的称为含硫化氢油气藏,5%~20%的称为高含硫化氢油气藏,20%以上者称为特高含硫化氢的油气藏,80%~90%以上者称为“纯”硫化氢藏。

实验原理

在一定的条件下,高含硫气体中 H_2S 可与水形成结晶水合物。从理论上讲,纯 H_2S 形成水合物的临界温度为 29℃。当天然气中 H_2S 含量超过 30% (摩尔分数) 时,生成水合物的温度基本上与纯 H_2S 相同。以下表 1 是根据理论和图表研究得出的不同压力下纯 H_2S 水合物的生成温度。

表 1 H_2S 水合物的形成温度

压力 (MPa)	0.10	0.11	0.12	0.14	0.17	0.72	1.62
温度 (℃)	0	1	2.1	3.4	5.2	19.8	28.5

由表 1 可以看出:纯 H_2S 形成水合物的温度相当高,而且随着压力升高还要逐渐升高。从生产角度出发,在如此高的温度下就形成水合物对气藏开发是相当不利的,这就使我们不得不开展对高含硫气藏水合物形成的机理和条件的研究。

对单组分气体的实验研究表明:在同一温度下,当气体蒸汽压升高时,形成水合物的先后次序分别是硫化氢 异丁烷 丙烷 乙烷 二氧化碳 甲烷

氮气。既然 H_2S 最容易形成水合物,那么对于高含硫气藏水合物形成机理的理论目前就有两种倾向。一种认为高含 H_2S 和 CO_2 气藏水合物的形成应按纯气体形成水合物的先后秩序,优先选择决定的。即首先是 H_2S 气体分子填充水分子的晶格,全部充填完后,再是异丁烷,最后是甲烷。另一种理论则认为水合物形成是按照天然气的比例以混合物整体的形式与水分子结合形成水合物,而不分先后顺序的。究竟哪一种理论更符合实际,有待实验来进一步研究。

首先,我们从逆向思维的角度来思考这个问题,设计相应的实验原理。假如第一种理论成立,在水

足够多的情况下,反应前后气体组分差异是相当大的,反应后的气体中有可能不含 H_2S 或 CO_2 这类容易形成水合物的气体。同样假如第二种理论成立,那么反应前后气体组分在色谱分析误差范围内应该是相同的,且水合物中气体组分的摩尔比例也应该相同。因而通过实验检验反应前后气体组分的变化情况,就能判断出高含硫气藏水合物形成的机理。

实验仪器

实验装置结构如图 1 所示,它是由 JEFRI 变体积高压蓝宝石全透明釜、搅拌系统、恒温空气浴、增压系统、温度压力测量系统、CCD 图像检测系统以及数据采集系统等组成。

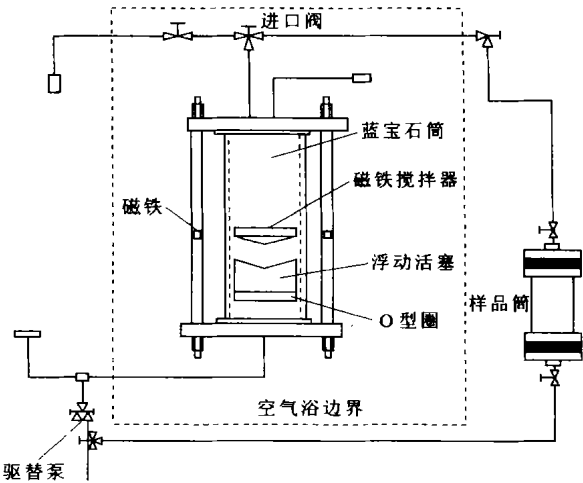


图 1 水合物实验装置流程图

实验方法

我们选取坡 4 井分离器气为气相,其组分组成见表 2,气体体积约为 60 mL。液相为坡 6 井的现场水样,水样必须为 H_2S 的饱和水样。因为在 1 atm 和 20℃ 下, H_2S 在水中的溶解度为 $2.58\text{ m}^3/\text{m}^3$,是相同条件下 CO_2 溶解度的 3 倍, CH_4 溶解度的 78 倍多。一旦气体与水接触后, H_2S 首先溶解于水中达到饱和后,剩余的 H_2S 才与水结合形成水合物,这就影响到了反应前后 H_2S 组分分析的计量,进而对实验结果分析产生较大的影响。参与反应的水样体积约为 3~5 mL。工作介质是质量比为 1:1 的乙醇水溶液。

实验中,通过温控装置控制初始温度为 38℃,从接口处进水样,安装好气样进出口管线,将蓝宝石筒紧固在恒温空气浴的水合物装置上。然后打开进气阀进气样,保持气体压力在 8 MPa,关闭进口阀,

启动磁性搅拌器,控制速度为 16 次/min 左右,最后通过冷冻装置开始逐级降温。实验过程中,我们采用定压测量,压力的控制通过改变反应筒的容积来实现。一旦水合物完全凝固形成后,通过进泵保持压力不变,打开进口阀,用已抽成真空的取样器取出蓝宝石筒剩余气体进行组分分析,判断反应前后气相组分的变化情况。

实验现象

在完成坡 4 井水合物生成的实验过程中,我们清楚地观测到:当温度降至 21.8 ℃ 时,磁铁搅拌速度明显变慢,水的粘滞阻力明显增强。一旦温度稍微下降,达到水合物生成的临界点,此时压力有一个明显的突降,通过釜壁可依稀观测到细小的白色颗粒,而 CCD 图像检测系统上明显观察到有晶核形成,并分散在液相中。这时稍微增大搅拌速度或降低温度,通过 CCD 图像系统可观测到,分散在液相中的晶核越来越多,并且不断聚结,出现了结晶状沉淀。此刻釜内液相已变得浑浊并且迅速堆积凝固,形成像冰块状的透明固体,堵塞了整个高压釜,这就是我们常说的天然气水合物。

实验结果

取出反应后残余气体进行组分分析,通过色谱数据得出反应前后气相组分组成变化见表 2。

表 2 坡 4 井水合物形成实验研究前后气样组分组成								
组 成	N ₂	H ₂ S	CO ₂	C ₁	C ₂	C ₃	iC ₄	nC ₄
反应前含量 (摩尔分数)	0.95	16.05	5.78	77.17	0.04	0.01	/	/
反应后含量 (摩尔分数)	0.96	16.25	5.70	77.04	0.04	0.01	/	/

同时,我们又针对 H₂S 含量较低的气样进行了上述实验研究,实验方法和实验步骤相近,其反应前后气体组分组成变化见表 3。

表 3 低含硫气体水合物形成实验研究前后气样组分组成								
组 成	N ₂	H ₂ S	CO ₂	C ₁	C ₂	C ₃	iC ₄	nC ₄
反应前含量 (摩尔分数)	1.28	1.73	5.40	91.53	0.05	0.01	/	/
反应后含量 (摩尔分数)	1.24	1.82	5.34	91.55	0.04	0.01	/	/

由以上两种气样样品分析结果可知:在色谱分析误差范围内,反应前后其组分几乎没有变化,这进

一步说明了在实验条件下,高含硫气藏水合物形成并非分步进行,而是以混合物整体形式同时反应生成的。实验中 H₂S 没有首先填充水分子空穴与其形成水合物,它只是促进气体水合物更容易生成。

结论与认识

通过上述实验研究,再结合部分理论资料,我们可以认为:在我们的实验研究范围内第二种理论是正确的。也即高含硫气藏水合物形成应按照天然气的比例以混合物整体的形式与水分子结合同时形成的,而不是按优先选择原则——H₂S 水合物首先形成的,实验前后气样组分的摩尔比例几乎不变。对高含硫气藏水合物形成机理的进一步研究,可以使

我们充分认识水合物形成的过程,从而有效地指导开发生产和地面工程建设。

另外,要对高含硫气藏水合物形成作更深一步的了解,不仅要对其形成机理做深入的研究,同时必须对水合物的生成条件进行系统的实验研究和测试。由于取样样品有限,同时 H₂S 有剧毒和腐蚀,因而也限制了我们实验数据的数量。目前,我们已完成了坡 4 井水合物生成条件的测试:在 8 MPa 下,水合物形成温度为 21.8 ℃;6 MPa 下,水合物形成温度为 20.1 ℃,而软件预测出水合物形成温度分别为 24.4 ℃ 和 22.5 ℃。对于最近才取回的罗家寨 7 井样品也进行了水合物形成条件的分析:H₂S 含量为 9 % 左右,在 8 MPa 下,水合物形成温度为 16.5 ℃;10 MPa 下,水合物形成温度为 18.0 ℃。对于低含硫气藏我们同样也进行了同样的测试,H₂S 含量为 1.8 % 左右的天然气,水合物在 8 MPa 下的形成温度为 11.5 ℃,而软件预测为 12.8 ℃。软件预测与实测值仍存在着一定的差别。究其原因,一方面因为天然气水合物成核、堆积、结晶是一个相当漫长的微观过程(可能长达几个小时或十几个小时),实验过程中往往由于时间有限不能精确观察和判断生成水合物的临界温度,因而实验所测数据比软件预测的数据低一些。另一方面,软件计算完全是根据状态方程和热力学理论推导而出的,而与实际结果存在着较大的差别,这主要是因为水合物的形成与气体组分组成、地层水离子浓度、气体扰动、杂质含量、酸性气体含量均有关系,而理论计算未考虑到这些影响因素。至于研究水合物形成的影响因素,我们曾作过专门的实验和研究(见《天然气工业》2002 增刊)。

由实验测试结果和理论研究我们可以得出:高含硫气藏在常温下极易形成水合物,给气藏开发造

车用轻烃油与油公核磁共振传递剂

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堀尾忠正等. 车用轻烃油与油公核磁共振传递剂. 天然气工业, 2003; 23(3): 100 ~ 104

摘 要 轻烃通过添加万分之二的油公核磁共振传递剂, 即可适合车用。但轻烃的密度不同, 使用方法也有区别: 对于密度在 0.67 kg/m^3 以上的轻烃, 通过添加油公万分之二后可直接供汽油车使用; 对于密度在 $0.64 \sim 0.65 \text{ kg/m}^3$ 的轻烃, 添加油公万分之二后按轻烃 30%、汽油 70% 比例混合使用; 对于密度为 $0.62 \sim 0.63 \text{ kg/m}^3$ 的轻烃, 则要在汽油车上另外加装一套由液态轻烃转化成气态轻烃进入发动机作功的转化系统。车用轻烃油与汽油相比: 动力不下降, 与汽油相当, 单耗比汽油下降 3% ~ 5%, 尾气中有害物质含量下降 90% 以上, 排放指标达欧、欧 标准。成品车用轻烃油零售价比汽油便宜 0.50 元/升以上, 与车用液化气价格相当, 具有优异的经济效益和环保效益, 并使石油资源得以充分利用。油公核磁共振传递剂是一种新型的物理性燃油、燃气添加剂, 添加后能提高热值、降低尾气排放量、增大动力、改善油品质量, 适用于一切液体和气体燃料。

主题词 轻烃 车辆 燃料 核磁共振 液体燃料 添加剂 热效率 废气排放标准

本文所述车用轻烃油是以 C_5 为主的轻烃作为原料加工而成的。轻烃的原料来源十分广泛, 主要来源于油田、气田、天然气净化厂等生产过程中的伴生气凝析液和炼油厂、溶剂油厂、石油化工厂、乙烯工厂的副产品, 如炼油厂生产的拔头油、轻石脑油、

溶剂油厂生产的石油醚, 油(气)田生产的稳定轻烃、天然气净化厂生产的“塔底油”等, 上述原料都能作为轻烃原料。

与其他燃料相比, 轻烃具有较高热值(比液化石油气热值要高出许多), 对人体没有毒害, 对环境几

成了相当大的困难, 因而必须确定水合物的形成机理和条件, 然后通过使气体温度维持在水合物生成的临界温度以上, 才能有效地防止生产油管及输送管线的堵塞。目前虽然我们的实验数据还很有限, 但随着高含硫气藏的不断开发, 样品数量的不断增加, 我们会研究出形成水合物的一系列实验数据, 包括不同温度, 不同压力, 不同含硫量和组分, 以此来指导具体的实际生产, 从而更有效地提高天然气田的采收率。据了解, 现今国外在处理和解决高含硫气藏水合物堵塞问题时有两种典型的处理方法: 一种是将适量的溶剂(热油溶剂)连续泵入井内油管和环行空间, 然后再借井口双通节流加热器进一步加

热, 防止水合物生成。另外一种是在下双油管, 注热油循环防止高含硫气藏中水合物的生成。

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ABSTRACT: A multiparameter model of calculating the filtration rate of fracturing fluid in dual porosity media is set up in the paper. Not only the influence of various experimental parameters (filter cake experimental pressure difference, fracturing fluid viscosity in experiment, the filtrate coefficient of the filter cake before stabilization and that after stabilization, etc.) on filter loss but also the influence of the three zones (filter cake zone, invasion zone and reservoir zone) of fracturing fluid filtration in real formation on filtration rates were considered in the model and the equation of expressing the filtration rate of fracturing fluid at reservoir zone under the control of dual porosity media percolation mechanism was derived by the authors. The changes in the pressures and filtration rates of the fracturing fluid at the three filtrating zones with times were clearly found through solving the simultaneous equation group of expressing the filtration rates at the three zones. The pressure changes and filtration rate changes at various filtrating zones in dual porosity media reservoirs and homogeneous reservoirs were contrasted and analyzed, which provides a theoretical basis for calculating the filtration rates in naturally fractured reservoirs.

SUBJECT HEADINGS: Low permeability pool, Fractured reservoir, Fracturing fluid, Multiple media, Filtration control, Mathematical model

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THE PREDICTION METHOD FOR WAX PRECIPITATION*

Mei Haiyan, Zhang Maolin, Li Shilun, Sun Liangtian, Sunlei, and Guoping (Southwest Petroleum Institute). *NA TUR. GAS IND.* v. 23, no. 3, pp. 92 ~ 94, 5/25/2003. (ISSN1000 - 0976 ; In Chinese)

ABSTRACT: Wax and asphalt solid precipitation in oil and gas recovering process is a problem considering very much at home and abroad in the petroleum industry. The organic solid precipitation happening in the recovering process along with the changes of thermodynamic conditions, such as temperature, pressure and composition, influences the oil and gas production severely. Especially in the process of gas injection for enhancing oil recovery, the wax and asphalt solid precipitation resulting from molecular transferring among phases may change permeability and wetability in the porous media and influence the improvement of recovering rate and the effect of gas injection. The article uses the state equation to describe the phase behavior of both gas and liquid phases, and the solution theory to correct the non-ideality of the solid phase. Then a thermodynamic model of gas-liquid-solid three phases is developed combining the gas-liquid balance model basing on the state equation and the liquid-solid balance model basing on the solution theory. The gas-liquid-solid three phases balance model is used to study the wax solid precipitation for an oil and gas system. The calculation results demonstrate that the state conversion of gas-liquid-solid

three phases happens as temperature and pressure changes. Pressure affects both the wax precipitation temperature and the amount of wax precipitation. The influence of pressure on wax precipitation temperature is much higher when the pressure is lower than the saturation pressure.

SUBJECT HEADINGS: Paraffin wax, Formation temperature, Miscible phase, Phase equilibrium, State equation, Saturation pressure

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TECHNIQUES OF SULFUR RECOVERY AND TAIL GAS PROCESS FOR GAS WITH HIGH SULFUR CONTENT*

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ABSTRACT: Gas fields with high sulfur content will be developed, and big gas treatment plants will be set up in Northeast Sichuan area for the project of 'East forward' gas transmission. Now, 2 sets of $300 \times 10^4 \text{ m}^3/\text{d}$ gas purification plants are going to be established in the area. The H_2S and CO_2 contents of their feed gas reach 10 % and 6 % respectively. The potential sulfur content reaches 800t/d, which is the most in China. According to the Chinese environment law, the tail gas discharging is allowed only if the sulfur recovery rate reaches above 99.7 %. So, severe requirements are made for the optimized design of the sulfur recovery and tail gas process plants. Introducing the new advance of the foreign techniques for sulfur recovery and tail gas process, integrating the technical exchanges with foreign companies and aiming to the general flow chart made from the preliminary feasibility study, the article recommends the proposal to utilize the techniques, such as two-stage Claus, cascaded Scot, and oxygen enrichment process, etc. in order to achieve the targets of higher quality of products, greater reliability in operation, and lower capital and operating costs.

SUBJECT HEADINGS: Sour gas, Sulfur recovery, Claus method, Scott method, Tail gas treating, Technology design

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EXPERIMENTAL INVESTIGATION OF HYDRATE FORMING MECHANISM IN GAS RESERVOIRS WITH HIGH SULFUR CONTENT*

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ABSTRACT: Gas reservoirs with high sulfur content are found one by one in LuoJiazhai, Tieshanpo, etc. of Northeast Sichuan area. Aiming to the hydrate forming mechanism of gas reservoirs with high sulfur content, the experimental investigation was conducted. The article discusses the experimental principles, methods, and phenomena in detail. It was demonstrated under the experimental conditions the hydrate formation of gas reservoirs with high sulfur content was according to the mechanism of the whole gas mixture combining with the water molecules but not the optimal principle one by one combining.

SUBJECT HEADINGS: Sour gas, Natural gas, Hydrate, Hydrogen sulfide, Forming mechanism, Forming conditions

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LIGHT HYDROCARBON OIL FOR VEHICLES AND TRANSFER AGENT OF YOUNGONG NUCLEAR MAGNETIC RESONANCE (NMR)

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ABSTRACT: Adding 0.02 % of transfer agent of Youngong NMR, light hydrocarbon can be used for vehicle's fuel. But the light hydrocarbons will be used with different ways according to their densities. As for the light hydrocarbons with the densities of more than 0.67 kg/m³, they can be used directly for vehicle's fuel by adding 0.02 % of transfer agent of Youngong NMR. As for the light hydrocarbons with the densities between 0.64 and 0.65 kg/m³, they can be used by mixing gasoline after adding 0.02 % of transfer agent of Youngong NMR. The light hydrocarbons will account for 30 % and the gasoline for 70 % in the mixture. As for the light hydrocarbons with the densities between 0.62 and 0.63 kg/m³, a conversion system, which can convert the liquid light hydrocarbons into gas will be installed for the vehicles using gasoline. Comparing with gasoline, the light hydrocarbons oil used for vehicles decreases 3 % ~ 5 % on consumption at the same power. The content of objectionable impurities in the tail gas decreases more than 90 %. The discharge index meets the standards of Europe and. The price of the light hydrocarbon oil used for vehicles is above 0.50 Yuan/L cheaper than that of gasoline, and is equivalent with that of liquid gas used for vehicles. So it has excellent benefit on economy and environment, and makes good use of petroleum resource. The transfer agent of Youngong NMR is a new physical additive for fuel oil and gas. It can increase the heating value, decrease the discharge value of tail gas, enhance the power and improve the fuel performance. It is applicable for all liquid and gas fuels.

SUBJECT HEADINGS: Light hydrocarbon, Vehicle, Fuel, Nuclear magnetic resonance (NMR), Liquid fuel, Additive, Thermal efficiency, Waste gas emission standard

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RELIABILITY OF IMPORTED LNG DELIVERING PROGRAM IN ZHUJIANG DELTA AREA *

Wu Zhongji (Baling Petrol-chemical Com.). NATURAL GAS IND. v. 23, no. 3, pp. 104 ~ 105, 5/25/2003. (ISSN1000 - 0976 ; In Chinese)

ABSTRACT: The density of population is big, the industrial agricultural and commercial sectors are highly developed, and the fuel demand is great in Zhujiang delta area. Since big oil and gas resource hasn't been found in this area, the fuel mainly comes from imported LPG for the past long term. Guangdong LNG receiving station which is going to set up will receive liquid natural gas (LNG) importing from Australia, etc. The project will make this area to use natural gas as main fuel. The paper introduces the gas delivering program for the imported LNG in Zhujiang delta area, compares the program of 'Reliable Round Net Gas Delivering System' based on the system reliability classification with the program of conventional branch gas delivering, and concludes that the 'Reliable Round Net Gas Delivering System' can deliver gas to the customers even under system repairing because of accidents. The reliability of the gas delivering system can be guaranteed. The system can deliver gas in a large scope and meet the long-term gas demand in this area. So, it is the optimum gas delivering program in Zhujiang delta area.

SUBJECT HEADINGS: Pearl river, Delta, Import trade, Liquefied natural gas (LNG), Gas supply, Project, Reliability

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APPLICATION OF HYDROCARBONS IN REFRIGERATION INDUSTRY *

Ma Zhenjun, Yan Gang, Zhou Jin and Wu Yezheng (Refrigerating & Cryogenic Engineering Department of Xi'an Jiaotong University). NATURAL GAS IND. v. 23, no. 3, pp. 106 ~ 109, 5/25/2003. (ISSN1000 - 0976 ; In Chinese)

ABSTRACT: The materials such as freon which are applied widely in the refrigeration industry will damage the ozone sphere